University of Alberta

The Feasibility of Coaxial Time Domain Reflectometry as an Insitu Site Characterization Tool for Determining the Moisture Content of Mine Tailings

by

Michel Elzear Lefebvre

A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of Master of Science

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research for acceptance, a thesis entitled **The Feasibility of Coaxial Time Domain Reflectometry as an Insitu Site Characterization Tool for Determining the Moisture Content of Mine Tailings** submitted by **Michel Elzear Lefebvre** in partial fulfillment of the requirements for the degree of Master of Science in Geotechnical Engineering.

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There is really nothing you must be.
And there is nothing you must do.
There is really nothing you must have.
And there is nothing you must know.
There is really nothing you must become.
However. It helps to understand that fire burns,
and when it rains, the earth gets wet...

Robert Fulghum

Abstract

The Athabasca oil sands deposit, located in northeastern Alberta, represents in excess of 15% of Canada's oil production. The dominant byproducts of the oil extraction process are coarse sand tailings and fine tailings. The fine tailings (composed of approximately 85% process water by volume) are deposited into a tailings basin and pose long term stability and reclamation issues. In order to implement new dewatering techniques, it is desirable to determine the solids content of the fine tailings in the basin. The current methodology for determining solids content involves removing samples from the basin for thermogravimetric analysis. The objective of this research project was to assess the feasibility of time domain reflectometry (TDR) as an alternative means for determining the insitu solids content of mine tailings. The theory of time domain reflectometry is reviewed. A Windows based TDR waveform analysis algorithm was developed for calculating the apparent dielectric constant of soil media. Issues related to pore fluid chemistry, probe geometry and coaxial cable length are investigated. A calibration protocol for fine tailings and recommendations for the development of calibration curves are discussed. Finally, a preliminary probe geometry for a push technology TDR probe is proposed.

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List of Symbols and Abbreviations

c	- speed of light
C	- response field capacitance
C_e	- emitter capacitance
C_r	- receiver capacitance
F	- response frequency
H	- inductance
j	- $\sqrt{-1}$
K'	- real dielectric constant
K"	- dielectric loss
K^*	- complex dielectric constant
Ka	- apparent dielectric constant
L	- length of probe
La	- apparent probe length
N	- refractive index
tanδ	- electric loss tangent
t°	- temperature (Celsius)
Vp	- velocity propagation constant
V_R	- reflected voltage
V_T	- transmitted voltage
w	- water content
Z_c	- impedance of coaxial cable
Z_o	- characteristic impedance of probe
Z_p	- impedance of probe

α - attenuation coefficient

ε_a - dielectric constant of air

ε_{bw} - dielectric constant of bound water (liquid)

ε_c - composite dielectric

ε_i - dielectric constant of ice

ε_o - free space permitivity

ε_s - dielectric constant of soil

ε_w - dielectric constant of water (liquid)

Γ - complex ratio of the reflected voltage to the incident voltage

ρ - dry soil bulk density

ρ_s - particle density

ω - angular frequency

σ - bulk soil electrical conductivity

σ_{dc} - DC conductivity

θ_v - volumetric water content

η - porosity

Chapter 1
Introduction

1.1 Statement of problem

The Athabasca deposit, located in northeastern Alberta, is the largest oil sands deposit in the province and currently represents in excess of 15% of Canada's oil production. By mass, typical Athabasca deposit oil sand ore is composed of 72% quartzose sand, 12% fines, 11% bitumen, and 5% water (Caughill et al., 1993). Its estimated bitumen reserves are approximately 869 billion barrels with 10 % of the deposit amenable to surface mining (FTFC, 1995).

The extraction process currently utilized by the two oil sands plants (Syncrude Canada and Suncor) is referred to as the Clark Hot Water Process. This extraction process separates the bitumen from the oil sand utilizing a combination of hot water, steam, and caustic soda (NaOH). The dominant byproducts of this process are coarse sand tailings, and fine tailings.

Perhaps the largest design issue from an economic, environmental and geotechnical perspective is related to the management and disposal of the fine tailings. Unlike coarse tailings, fine tailings (composed of 85% water by volume) do not settle rapidly and therefore pose long term stability and reclamation issues (List and Lord, 1995). After deposition in the settling basin, fine tailings dewater relatively rapidly for the first two to three years after which time the solids concentration reaches a value of approximately 30%. When the fine tailings reach this stage, the suspension develops non-Newtonian properties and has a consistency somewhat like yogurt (Sheeran, 1993). Further increase in solids concentration / dewatering occurs very slowly, therefore, several hundred years or more may be required for the fine tailings to develop the strength of a soft clay.

The amount of fine tailings produced by the extraction process is a function of the ore quality. At Syncrude, 0.18 tons of fine tailings are produced per ton of oil sand ore. This translates to an approximate annual accumulation of 15 - 20 million cubic meters of fine tailings. Currently, in excess of 275 million cubic meters of fine tailings have been produced with an estimated lifetime (under the current lease) accumulation of 800 - 1000 million cubic meters. Hence, the volume of semi-fluid fine tailings is of considerable concern (MacKinnon and Sethi, 1993).

Syncrude incurs significant operating costs is due to the construction of fine tailings containment dykes, and pumping systems. Therefore, Syncrude has spent considerable effort to develop new techniques to reduce the total volume of fine tailings being produced. One such technique is the development of nonsegregating or combined tailings. This method enables in excess of 95% of the fines to be retained in the coarse tailings stream (Caughill et al., 1993), hence enabling significantly higher solids contents. Therefore, the tailings are able to attain higher rates of stability in a significantly shorter time span.

Research is currently underway to determine the most effective and economically feasible method for disposing of fine tailings, and to accelerate the rate at which dewatering occurs. However, in order to implement any advancement in dewatering methodology, it is necessary to first characterize the current tailings basin. One such characteristic is the solids content in relation to 3-dimensional space. Current methodology for measuring water content consists of lowering a "corked jar" into the basin and pulling the cork at the desired depth. The jar is then raised to the surface and the water content determined via gravimetric analysis. This technique may be problematic due to the potential for contamination of the contents of the unsealed jar as it is raised to the surface, and the high degree of disturbance to which the sample is subjected.

1.2 Thesis Objective

The objective of this thesis involved the assessment of time domain reflectometry (TDR) as an alternative means for determining the solids content of mine tailings.

The main objectives of this research project are as follows:

- ⇒ explore the principals of TDR;
- ⇒ assess the feasibility of TDR for use in the characterization of mature fine tailings (MFT);
- ⇒ produce a field system which would permit the insitu solids content characterization of mine tailings.

Chapter 2

Literature Review

2.1 Introduction

The following literature review was conducted to determine the most feasible method for measuring moisture content in mine tailings such as the mature fine tailings (MFT) and combined tailings (CT) present at Syncrude Canada. The format of this literature review can be perceived as a "road map" for future work in this area. Section 2.5 is a brief synthesis of the literature review and explanation for the selection of time domain reflectometry for this research project. As a final note, the path taken on a journey is often more important than the actual arrival at one's destination. I hope this review will aid in finding a better route.

2.2 Methods for determining water content

Soil water content is possibly the most tangible soil property and is fundamental to site investigations in the sciences of agriculture, forestry, hydrological and civil engineering. The definition of "soil water content" is widely accepted as the amount of water which will evaporate from a soil by heating a sample to approximately 105°C. This definition is often utilized to calibrate other methods. Caution should be exercised in accepting this definition. As stated in Gardner (1965):

> *"the choice of the particular temperature range appears not to have been based upon scientific consideration of the drying characteristics of soil".*

In excess of 38 methods for measuring soil moisture content are currently known (Gringof and Nabiev, 1987). These methods may be classified as either direct or indirect soil moisture measurements.

2.3 Direct methods for determining water content

Direct methods are defined by removal of pore water from the soil via evaporation. The principal direct method is referred to as the thermogravimetric method. This method is relatively simple and therefore the most widely accepted and frequently utilized (Topp et al., 1996). A soil sample is weighted and placed in an oven at a temperature of approximately 105°C for 24 hours or until constant mass is achieved. The gravimetric water content (w) of the sample is a mass to mass relationship which is calculated by dividing the mass of the water by the mass of the soil solids. The following equation published in Craig (1992) is commonly used to calculate gravimetric water content:

$$w = \frac{\text{mass of water}}{\text{mass of dry soil}} \qquad (2.1)$$

From the gravimetric water content, the volumetric water content (θ_v), a volume of water to volume of soil relationship, may be calculated. Gravimetric water content is typically converted to volumetric water content to calibrate indirect water content measurement technologies. Volumetric water content may be calculated, as a function of gravimetric water content (w) and dry soil bulk density (ρ) utilizing the following equation (Smith and Mullins, 1991):

$$\theta_v = w \rho \qquad (2.2)$$

The principal disadvantages of direct water content measurements include:

- ⇒ the need to remove soil samples from the site;
- ⇒ the destructive nature of the test procedure;
- ⇒ the inability to replicate the test procedure on a single sample;
- ⇒ the labor intensive nature of the test procedure which requires 24 hours to complete.

2.4 Indirect methods for determining water content

Indirect methods for determining water content may be defined as those techniques which are dependant upon a measurable soil property which is a function of water content (Smith and Mullins, 1991). These methods rely on the placement of instrumentation either within or on top of the soil, or via remote sensing. Two soil properties are commonly utilized: hydrogen nuclei concentration and dielectric constant.

2.4.1 Hydrogen nuclei

Hydrogen nuclei detection systems developed for indirect measurement of water content are commonly referred to as neutron probes. This method utilizes the ability of hydrogen to decelerate fast neutrons. The fundamental principals of the neutron probe and its operation are discussed in Gardner and Kirkham (1952). The neutron probe is composed of two parts: a high energy neutron emitter and a slow neutron detector. The neutron probe is operated in soil by lowering the emitter / detector unit down an aluminum access tube which has been inserted in a borehole. Figure 2.1 illustrates a typical neutron probe. Spatial sensitivity is discussed in Smith and Mullins (1991) and Chanasyk and Naeth (1996).

High energy neutrons are emitted from a radioactive source and are slowed by elastic collisions with the nuclei of atoms - particularly hydrogen nuclei. This process is referred to as thermalization (Chanasyk and Naeth, 1996). The detector counts the number of slow neutrons which return to the probe per unit time. The relative number of slow neutrons is referred to as the "count rate" (Smith and Mullins, 1991).

Based on the premise that most of the hydrogen present in soil is due to water content, a calibration curve may be produced relating "count rate" to volumetric water content. This calibration is generally believed to be a linear relationship for volumetric water contents less than 40% (Merriam and Knoerr, 1961).

Manufacturers of these probes supply a calibration curve which may prove unreliable for many applications, therefore, field or laboratory calibrations may be required (Rawls and Asmussen, 1973), (Silvestri et al., 1991), and (Morris and Williams, 1990).

Negative aspects of the neutron probe methodology arise due to neutron thermalization. Silvestri et al. (1991) note the factory calibration is inadequate for soft clays and high water contents. The authors also state that calibration curves may be soil specific in view of the effect of bulk density, mineralogy, texture, and salinity on neutron thermalization. Figure 2.2 illustrates a set of calibration curves for use in the high water content sensitive clays of Quebec. This figure clearly demonstrates the difficulties which arise in accurately measuring volumetric water contents greater than 40%.

2.4.2 Dielectric constant

The dielectric constant of a material is due to its polarization - the alignment of the dipoles. Hence, the dielectric constant may be defined as the ability of a material to act as an insulator or the capacity of a material to reduce conductance of electromagnetic energy (Kaya et al., 1994). The physics of dielectrics is reviewed in Boylestad (1990). The concept of dielectrics is clearly defined by Smith and Mullins (1991):

> *"A practical definition of the dielectric constant K of a material is the ratio of the value of a capacitor with the material between the plates, compared with the value with air between the plates. A dielectric material is an insulator, as distinct from a metal, which is a conductor. Under the influence of an electric field, the positive and negative charges in a dielectric material are displaced with respect to each other and tiny electric dipoles are produced. Some materials, such as water, also have permanent dipoles. The electric dipoles are*

aligned by the electric field, and the dielectric medium as a whole becomes polarized. The dielectric constant as defined above turns out to be a measure of the polarization; as a consequence, a material whose molecules have a permanent dipole moment are free to align with the electric field has a very large dielectric constant."

This definition is a useful aid for comprehending why the dielectric constant of a soil media is approximately 4.5 (variations between 3 - 6 have been noted in the literature), the dielectric constant of air is 1, and the dielectric constant of distilled water at 20°C is approximately 80. Water molecules are more readily polarized than the molecules which comprise soil or air, therefore, the dielectric constant of water is significantly higher. Likewise, the dielectric constant of water varies with temperature and phase. While in the liquid state, water molecules polarize readily. However, in the solid state (ice) the molecules cannot readily align, therefore, a dielectric constant of approximately 3.2 is typically measured.

Due to the large variation in dielectric constant between soil and water (aqueous phase) it is possible to measure the dielectric constant of a soil-air-water system to determine soil moisture content. Balygin and Vorob'yev (1934) were among the first to note that the dielectric constant of soil increases with increased water content. However, the dielectric constant of a soil media is also affected by the concentration of soluble salts, density, and frequency. Of these factors, the most important is frequency (Kaya et al., 1994 and Selig and Manusukhani, 1975). As illustrated in Figure 2.3, at low frequency ranges less than 30 MHz and at high frequency ranges greater than 3 GHz, the measure of dielectric constant for a soil medium varies significantly. The reasons for this phenomena are discussed further in Smith and Mullins (1991), Hoekstra and Delaney (1974), and Davis and Annan (1977).

The complex dielectric constant (K^*) may be expressed by the following equation (Davis and Annan, 1977):

$$K^* = K' - j\,(K'' + \sigma_{dc} / (\omega\,\varepsilon_o)) \qquad (2.3)$$

Where K' represents the real dielectric constant, K" represents the dielectric losses, j represents the square root of -1, σ_{dc} represents direct current (DC) conductivity, ω represents the angular frequency, and ε_o represents the free space permittivity.

In general, the real dielectric (K') and the imaginary dielectric (K") are functions of angular frequency (ω). The real part of the complex dielectric is an index of the electric flux density while the imaginary part describes losses due to conductivity. However, as illustrated in Figure 2.4, the complex dielectric constant is approximately constant and the imaginary dielectric constant is negligible for the frequency range of 30 MHz - 3 Ghz (Wobschall, 1978, HalliKainen et al., 1985, Topp et al., 1988, and Arulanandan, 1991).

Two principle technologies are currently utilized for determining the dielectric constant of a soil-water-air system: the frequency domain and the time domain. The frequency domain is a frequency specific technology which measures either capacitance or impedance while the time domain measures the velocity of electromagnetic waves over a wide frequency range.

2.4.3 Frequency domain

Dielectric constant measurements made in the frequency domain are by definition, frequency specific. Figure 2.5 illustrates the electromagnetic frequency spectrum. The practical range for analysis of soil is in the radio frequency range from approximately 30 MHz to several hundred MHz. In this range the measured dielectric of soil is constant.

Early work in the frequency domain was carried out at low frequencies in the kHz range by Smith-Rose (1933). Measurements at these frequencies gave unusually high values of K due to interficial polarization effects in heterogeneous materials such as moist soil (Hoekstra and Delaney, 1974). A considerable body of literature has been produced regarding dielectric measurements in soil over a frequency range encompassing DC through to the microwave spectrum. These papers are briefly reviewed in Campbell (1990) and include; Smith-Rose (1933), Paquet (1964), Scott et al. (1967), Hoekstra and O'Brien (1969), Lundien (1971), Geiger and Williams (1972), Cihilar and Ulaby (1974), Hipp (1974), Hoekstra and Delaney (1974), Selig and Mansukhani (1975), Davis and Annan (1977), Topp et al. (1980), Olhoeft (1985), Jackson (1990), Scott and Smith (1992), Dean et al. (1993), and Stroemich et al. (1994).

Techniques for determining the dielectric constant in soil utilizing the frequency domain can be broken down into two related methods: capacitance and impedance probes. Although these two technologies utilize very similar hardware and some authors use these terms interchangeably (Eller and Denoth, 1996), for theoretical considerations they will be dealt with separately.

2.4.3.1 Capacitance method

The capacitance method operates on the premise that a moist soil functions as part of the dielectric of a capacitor as illustrated in figure 2.6. The physics of capacitance are reviewed in Boylestad (1990).

The capacitance probe operates at a selected frequency within the aforementioned range of 30 MHz to several hundred MHz. The soil media surrounding the probe responds by oscillating at a similar frequency with some energy being stored in the soil due to capacitance, therefore, the responding frequency is less than the initiating frequency as (Tomer and Anderson (1995). The probe measures this frequency shift which is a function of water content due to the effect of water on

soil electrical properties. The relation between frequency and capacitance is described as (Tomer and Anderson (1995):

$$F = (1/C + 1/C_e + 1/C_r)^{1/2} / (2\pi H^{1/2}) \qquad (2.4)$$

Where the response frequency (F) is a function of response field capacitance (C), emitter capacitance (C_e), receiver capacitance (C_r), and inductance (H). The theory of operation of capacitance probes is further discussed by Wobschall (1978), Kuraz (1981), Dean et al. (1987), and Straub (1994).

A major advantage of the capacitance method is that it is amenable to a variety of electrode geometries and sizes. Several geometries which have been utilized for capacitance probes are illustrated in Figure 2.7 and Figure 2.8. Commercially manufactured capacitance probes are currently available. One such probe (Kuraz, 1981) is illustrated in Figure 2.9. The most notable difficulty related to the field use of this probe is the need for placement in an access tube similar to that utilized by neutron probes. Also of concern is the influence of soluble salt concentrations and temperature (Kuraz, 1981) which may significantly affect the shape of the calibration curve. Recent work (Eller and Denoth, 1996) has produced the capacitance measuring device illustrated in Figure 2.10. However, no data was available with regards to the effects of temperature and soluble salts.

2.4.3.2 Impedance method

The impedance probe functions on similar principles to the capacitance probe. The most significant difference between these two methods is the variation in probe geometry (Figure 2.11) as illustrated in Campbell (1990), Ungar et al. (1992), and Gaskin and Miller (1996). The probe utilized in this method produces an electromagnetic field similar to a coaxial cable. Voltage is applied to the inner tine via a coaxial cable which results in a well defined electric field volume. The

resulting probe impedance (Z_p) can be calculated by the following equation (Collin, 1966):

$$Z_p = Z_o / K^{1/2} \text{ cotanh}(i \omega K^{1/2} L/c) \quad (2.5)$$

Where Z_o is the characteristic impedance of the probe, K is the dielectric constant, L is the probe length, and c is the velocity of light.

The probe impedance is calculated by utilizing a network analyzer (Campbell, 1990) as illustrated in Figure 2.12. From the network analyzer, the complex ratio of the reflected voltage to the incident voltage (Γ) can be determined. The probe impedance (Z_p) is therefore calculated as a function of the impedance of the coaxial cable (Z_c) and Γ (Campbell, 1990):

$$Z_p / Z_c = (1 + \Gamma) / (1 - \Gamma) \quad (2.6)$$

The probe impedance calculated from equation 2.6 is substituted back into equation 2.5 to calculate the dielectric constant of the material.

2.4.4 Time domain

Time domain reflectometry (TDR) is a remote sensing technique which is commonly utilized to determine the spatial location of an object. The most familiar form of TDR, developed in the 1930's, is RADAR which is composed of a radio transmitter which emits a short pulse of microwave energy, a directional antenna, and a radio receiver (Andrews, 1994). Once a pulse is transmitted, the receiver "listens" for the reflected signal. By measuring the time delay between the transmission and reception of the reflected pulse, the distance to the object can be calculated.

This principal is also valid for coaxial TDR which is utilized to determine the dielectric constant of a material in which a probe is embedded. The TDR unit measures the time required for the signal to travel the length of the transmission lines (referred to as waveguides). The concept of TDR is based on the measurement of the dielectric constant of a medium by determining the signal propagation velocity in a transmission line which is independent of both the line geometry and the capacitance (Topp and Davis, 1985c). Figure 2.13 illustrates a typical TDR system for measuring soil moisture. The TDR unit emits a fast rising (< 200 ps) step pulse (Figure 2.14). A step pulse is utilized due to the long plateau which conveys direct current (DC) information regarding the reflecting object, while the fast rise time contains very high frequencies which provide good spatial resolution (Andrews, 1994). A typical frequency bandwidth is approximately 20 kHz to 1.5 GHz (Heimovaara, 1994). The transmission lines / waveguides, illustrated in Figure 2.13 act as conductors, while the soil acts as the dielectric medium. The fast rising step pulse emitted from the TDR unit is reflected from the end of the waveguides and returns to the TDR receiver producing a waveform (Figure 2.13). From this information the propagation velocity of the pulse through the soil can be calculated. The propagation velocity is indicative of the dielectric constant and therefore, the volumetric water content.

TDR fundamentals and theoretical considerations are reviewed in greater detail in Topp and Davis (1982a & b), Topp and Davis (1985a & b), Ledieu et al. (1986), Zegelin et al. (1990), Whalley (1993), Kaya et al. (1994), Topp et al. (1994) and White et al. (1994).

2.4.4.1 Early TDR research

The first widely cited publication on the measurement of dielectrics in the time domain was Fellner-Feldegg (1969). The author reports that dielectric measurements made for soil characterization with time domain reflectometry are similar to those made in the frequency domain. He concludes that the time domain

permits the same information to be collected in only a fraction of the time with less expensive equipment.

Davis and Chudobiak (1975) discuss the application of time domain reflectometry (TDR) to measure the electrical properties of soils. The propagation velocity (V) of a transmission line / waveguide can be determined by:

$$V = c / \{K'(1 + (1 + \tan^2\delta)^{1/2}/2)\}^{1/2} \qquad (2.7)$$

where $\tan\delta$ is the electric loss tangent defined by:

$$\tan\delta = \{K'' + (\sigma_{dc} / \omega\varepsilon_o)\} / K' \qquad (2.8)$$

In media with low electrical loss, $\tan\delta << 1$, the propagation velocity can expressed as;

$$V = c / (K')^{1/2} \qquad (2.9)$$

Davis and Annan (1977) expanded research into measurements of dielectrics utilizing TDR. From their test results, the authors produced the following conclusions:

⇒ The real part of the dielectric constant (K') is strongly dependent on soil moisture and only weakly dependent on other soil properties such as soil type, density, and temperature. The approximation of the apparent dielectric constant equal to the real part of the dielectric constant ($Ka \cong K'$) appears valid.

⇒ A variation of several hundred percent is observed for the apparent dielectric constant as a function of volumetric water content. The apparent dielectric constant is a sensitive indicator of moisture

content due to the high degree of polarizability of free water which swamps other soil property variations of Ka.

⇒ The dielectric loss component (K") appears to vary significantly with soil type. Noncohesive soils show little attenuation while cohesive soils show substantial attenuation of TDR signals.

The authors state that the apparent dielectric constant can be expressed as:

$$Ka \cong K' \cong (c / V)^2 \cong (ct / L)^2 \qquad (2.10)$$

The first practical application of TDR was published by Topp et al. (1980). The objective of this paper was to establish the dependence of Ka on the volumetric water content (θ_v) over the frequency range of 1 MHz to 1 GHz for a wide range of soils. The authors acknowledged the complexities of the electrical properties of saturated soils (Davis and Annan, 1977, Wobschall, 1977, and Wobschall, 1978) and recognized the need for empirical correlation. Tests were performed on clays, sands, vermiculite, and glass beads at volumetric water contents ranging from 0 to 55% and temperatures ranging from 10 to 35°C. The results of their study demonstrated that Ka is strongly dependent on the volumetric water content of the soil. In addition, Ka is almost independent of soil density, mineralogy, salt content, and temperature. The authors present a third order polynomial equation which fits their data. This equation is typically referred to as the Topp equation:

$$Ka = 3.03 + 9.30\theta_v + 146.0\theta_v^2 + 76.7\,\theta_v^3 \qquad (2.11)$$

2.4.4.2 Calibration for soil water content

Two approaches are presented in the literature to relate soil water content and dielectric constant for the calibration of TDR systems: functional relationships and dielectric mix models. Functional relationships are selected by their ability to fit

experimental data points (empirical relationships). Dielectric mix models relate the composite dielectric number of a multiphase mixture to the dielectric constant values and volume fractions of its constituents (Roth et al., 1990).

2.4.4.2.1 Soil water content calibrations above freezing temperatures

The functional relationship approach, utilized by Topp et al. (1980), demonstrated that a third order polynomial regression equation could be utilized to represent the relationship between apparent dielectric constant and volumetric water content. This equation (2.11) has been proven adequate for a wide range of agricultural soils with $\theta_v < 40\%$ (Skaling, 1990) and is essentially independent of soil bulk density, ambient temperature, and salt content. The Topp equation appears to be adequate for coarse textured cohesionless soils, but may be inadequate for fine textured, dense, heavy cohesive soils and organic soils (Zegelin et al., 1990, Roth et al., 1990, Roth et al., 1992, and Dirksen and Dasberg, 1993). Other empirical relationships have been developed such as that utilized by Soilmoisture Equipment Corp. (Skaling, 1990). The TRASE calibration illustrated in Figure 2.15 is a composite of several best fit curves and is valid over the entire range of moisture contents ($0 \leq \theta_v \leq 100\%$).

The increasingly widespread utilization of TDR for measuring soil moisture content has resulted in a number of applications where Topp's equation is inadequate (Topp et al., 1994). Hence, an alternative to empirical calibrations has been developed by deriving a calibration equation from dielectric mixing models. These models relate the dielectric constant of a multiphase mixture of soil, air, and water to the dielectric numbers and volume fractions of the individual components (Roth et al., 1990). Theory related to dielectric mix models is presented in Tinga et al. (1973), and Geyer (1988).

Roth et al. (1990) published a three phase mixing law to describe wet soil:

$$\varepsilon_c = (\theta\varepsilon_w^{\alpha} + (1 - \eta)\varepsilon_s^{\alpha} + (\eta - \theta)\varepsilon_a^{\alpha})^{1/\alpha} \qquad (2.12)$$

Where the volume fractions θ, $1 - \eta$, $\eta - \theta$ of water, soil, and air are linked to the corresponding dielectric constant of water (ε_w), dielectric constant of soil (ε_s), and dielectric constant of air (ε_a). The parameter α ($-1 \leq \alpha \leq 1$) is a geometric factor which is dependent on the spatial arrangement of the mixture and its orientation in the electric field (Topp et al., 1994). A typical value of $\alpha = 0.5$ is cited by Alharti and Lange (1987). For situations in which large variations in temperature occur, or high volumetric water contents, the dielectric constant of free water (ε_w) can be calculated as a function of temperature in degrees Celsius (Handbook of Chemistry and Physics, 1995):

$$\varepsilon_w = 78.54\,[(1 - 4.579 * 10^{-3}\,(t° - 25) + 1.19 * 10^{-5}\,(t° - 25)^2 - 2.8 * 10^{-8}\,(t° - 25)^3] \qquad (2.13)$$

For cohesive soils, the dielectric constant of both free and bound water may be taken into account with a four-phase mix model (Dobson et al., 1985, Bohl and Roth, 1994):

$$\varepsilon_c = (\theta_{bw}\varepsilon_{bw}{}^{\alpha} + \theta_{fw}\varepsilon_{fw}{}^{\alpha} + (1 - \eta)\varepsilon_s{}^{\alpha} + (\eta - \theta)\varepsilon_a{}^{\alpha})^{1/\alpha} \qquad (2.14)$$

This equation modifies equation 2.12 by dividing the volumetric water content fraction into free water (fw) and bound water (bw) components with the dielectric constant of bound water (ε_{bw}) having a value of approximately 3.2.

The current trend in TDR calibrations is to reduce the calibration equation for both empirical and mix model methodologies to a linear relationship (Topp et al., 1996):

$$Ka^{1/2} = C_1\,\theta + C_2 \qquad (2.15)$$

The square root of the apparent dielectric constant is referred to as the "Refractive Index" (Whalley, 1993). The constants C_1 and C_2 are dependent on the soil matrix.

Because this relationship is linear, the calibration is simplified by requiring only two points (Topp et al., 1996). This linear relationship is discussed in Herkelrath et al. (1991), Whalley (1993), Topp et al. (1994), White et al. (1994), Hook and Livingston (1996), Ferre et al. (1996) and Topp et al. (1996).

Current research is also focused on the effects of soil matrix and bulk density on dielectric constant versus volumetric water content relationship. The literature indicates that soil matrix and bulk density influence TDR readings. This effect can be reduced by either accounting for bulk density or porosity. This subject is further discussed by Jacobsen and Schjonning (1993), Malicki et al. (1994), White et al. (1994), and Malicki at al. (1996).

2.4.4.2.2 Calibration for unfrozen water content in frozen soil

It has been well documented that liquid water coexists with ice in soil at temperatures below 0 degrees Celsius (Anderson and Tice, 1972, and Seyfried and Murdock 1996). The relative amount of liquid water and ice affects infiltration rates in frozen soil, and freezing-induced entrained or dissolved chemical movement (Seyfried and Murdock, 1996).

Dielectric mix models can be utilized to derive TDR calibrations for unfrozen moisture contents by substituting the assumed dielectric constant for ice ($\varepsilon_i = 3.2$) into a four phase dielectric mix model. Reasonable results have also been achieved by utilizing Topp's equation (Seyfried and Murdock, 1996). Further discussion on the measurement of unfrozen water content and the development of TDR calibrations can be found in Hayhoe et al. (1983), Stein and Kane (1983), Mulla (1985), Topp and Davis (1985), and Spaans and Baker (1995).

2.4.4.3 Probe development

Time domain reflectometry transmission-line probes are commonly referred to as waveguides. The transmission line acts as a conducting medium while the soil acts

as the dielectric medium (Topp and Davis, 1985). When a change in impedance is encountered by a signal as it propagates along a transmission line, part of the signal is reflected back towards the source. It is critical to the accuracy of the system to minimize reflections and signal loss in the cable connecting the TDR unit to the probe, thereby maximizing the signal strength entering the soil and reflecting back to the receiver.

Waveguides are typically connected to the TDR unit via a 50Ω coaxial cable. The selection of probe geometry and cable length can significantly affect the accuracy of the system. The effects of coaxial cable type and length are presented in Zegelin et al. (1992), Heimovaara (1993), and Hook and Livingston (1995).

For the purpose of discussing probe geometry, TDR probes have been categorized as: coaxial probes, two wire probes, three / four wire probes, and other geometries.

2.4.4.3.1 Coaxial probes

The early TDR probes were referred to as "coaxial soil containers" as illustrated in Figure 2.16. This probe configuration, utilized by Topp et al. (1980) was designed for laboratory use and is not practical for field applications.

2.4.4.3.2 Two wire probes

The use of two wire parallel transmission lines is discussed in Topp et al. (1982a & 1982b). A typical parallel wire TDR circuit is illustrated in Figure 2.17. In order to maximize the signal strength entering the probe, an impedance balancing transformer (balun), with a broad-frequency bandwidth, is placed in the circuit between the coaxial cable and probe. Topp and Davis (1995) recommend utilizing the Anzac model TP103 impedance-matching transformer for this purpose. Further details regarding the use of baluns can be found in Spaans and Baker (1993).

Ledieu et al. (1986) produced a further advancement in parallel probe design. Their probe consists of two stainless steel rods, 5 mm in diameter, 25 mm apart, and 30 cm in length (Figure 2.18). The significant advancement in this probe was the incorporation of two opposing diodes. The purpose of these diodes was to accurately mark the beginning of the probe (the connection point between the coaxial cable and waveguides) with an artificial impedance discontinuity.

The effects of probe geometry on spatial resolution for parallel probes is discussed by Knight (1992). Figure 2.19 illustrates the dimensions of a parallel-wire probe. Knight recommends that the ratio of b/d should not be less than 0.1 and concludes that the rod diameter should be as large as possible compared with the rod spacing to minimize the high energy density "skin effect" around the probe rods. Petersen et al. (1995) suggests that that probes should be designed as long as practically possible. The geometry of parallel wire transmission lines is discussed further in Malicki et al. (1992), Kelly et al. (1995) and Maheshwarla and Venkatasubramanian (1995).

Ferre et al. (1996) explore the use of twin rode probes with dielectric coatings. Coated waveguides are useful for maximizing the useable length of TDR probes by minimizing energy losses which are dissipated due to electrical conduction. Recommendations are made by the authors with regards to probe design and selection of coating material.

2.4.4.3.3 Three and four wire probes

Zegelin et al. (1989) developed the three / four wire TDR probe geometry as an intermediary step between the coaxial transmission line cell and the two wire / parallel waveguide probe. The multi-wire probes (Figure 2.20) emulate coaxial transmission lines, therefore eliminating the need for impedance balancing transformers (baluns). Multi-wire probes are simple to construct, inexpensive, and provide clearer signals than twin rod probes. As illustrated in Figure 2.21, the three

and four wire probe geometries create an electric field similar to a coaxial cable, therefore minimizing the impedance mismatch which occurs at the coaxial cable - waveguide connection point. The wires are equi-distant and parallel to one another. If the probe wires are not parallel, the TDR signal may reflect changing impedance along the length of the probe (Zegelin et al., 1990). However, it has been demonstrated by Kachanoski et al. (1990) that the wires need not be straight. The design of triple-wire probes is discussed in further detail in Zegelin et al. (1990) and Heimovaara (1993).

Further design advances have been proposed by Hook et al. (1992) utilizing remote shorting diodes to maximize reflection detection of the beginning and end of a triple-wire TDR probe. The authors state that this technique permits cable lengths of approximately 100m to be utilized.

2.4.4.3.4 Other probe geometries

Several other probe geometries have been described in the literature for TDR applications for determining soil water content. Selker et al. (1993) presented a design of a non-invasive probe (NIP) utilizing serpentine parallel waveguides mounted on an acrylic pad (Figure 2.22). Yokuda and Smith (1993) presented a design for a probe which can be inserted to a depth of 14 feet (approximately 4.3 meters) into soil utilizing a vibratory drill (Figure 2.23). Ferre et al. (1994) presented a design for a multilevel waveguide to conduct water content profiling by TDR. Kaya et al. (1994) proposed a split spoon sampler for TDR use (Figure 2.24). Finally, Noborio et al. (1996) described a probe design capable of measuring water content, heat capacity, and thermal conductivity (Figure 2.25).

2.4.4.4 Spatial sensitivity of time domain reflectometry probes

Baker and Lascano (1989) addressed two aspects related to TDR probes which had not been clearly defined. These issues are the volume of soil affecting the measurement of soil moisture and the pattern of influence within that volume. Their

findings are illustrated in Figure 2.26. Data indicated that the sensitivity ends abruptly at the end of the waveguides. Utilizing 300 mm twin rod probes (b = 3.175 mm and d = 50 mm as illustrated in Figure 2.19) the authors conclude that soil moisture measured by TDR is largely confined to a quasi-rectangular area of approximately 1000 mm^2 surrounding the waveguides with no significant variation in sensitivity along the length of the waveguides.

Spatial weighting functions are discussed in Knight (1992). The author concludes that if the wire diameter (b) is small relative to the distance between the wires (d) then a "skin effect" occurs producing a high energy density around the wire. This "skin effect" will result in local soil conditions surrounding the wires to disproportionately affect the apparent dielectric constant. The author also concludes that 95% of the energy is contained within a cylinder radius of 2.582d when b = 0.1d. This cylinder increases in size to 2.900d when b = 0.2d.
Other work related to spatial sensitivity and weighting functions can be found in Knight (1991), Baker and Lascano (1991), Knight et al. (1994), and Ferre et al. (1996).

2.4.4.5 Electrical conductivity measurements using TDR

Transverse electromagnetic waves (TEM) are emitted from the TDR unit and propagate through the coaxial cable and the waveguides buried in the soil. The signal energy is attenuated due to the media through which the signal travels as illustrated in Figure 2.27. The degree of signal attenuation is proportional to the electrical conductivity of the material. The reduction in voltage as the signal travels through the soil media enables TDR to measure bulk soil conductivity (Hubscher et al., 1996).

Dalton et al. (1984) first demonstrated that bulk soil electrical conductivity (σ) could be measured simultaneously with water content utilizing a parallel rod transmission line (twin waveguide probe with balun). The authors assume perfect

signal reflection at the end of the waveguides. The reflected voltage (V_R) is expressed as a function of the transmitted voltage (V_T), the attenuation coefficient (α), an the waveguide length (L):

$$V_R = V_T \exp(-2\alpha L) \qquad (2.16)$$

Figure 2.28 illustrates how V_R and V_T are determined from the TDR waveform output, hence, the bulk electric conductivity can be calculated as (Dalton et al., 1984):

$$\sigma = \{(Ka)^{1/2} / (120\pi L)\} \ln(V_T / V_R) \qquad (2.17)$$

Although the methodology for calculating bulk soil conductivity was improved by Topp et al. (1988) utilizing the thin sample method presented in Giese and Tiemann (1975), the method described by Dalton et al. (1984) is considered valid for most geotechnical applications (Kaya et al., 1994).

Initial experimentation was performed utilizing twin rod waveguides with an impedance balancing transformer (balun). However, increased accuracy was achieved by utilizing triple-wire probes (Zegelin et al., 1989, and Nadler et al., 1991). The aforementioned technique (developed with twin waveguide probes) is also valid with triple and multiple waveguide probes.

Further literature related to bulk soil electrical conductivity can be found in Balygin and Vorob'yev (1934), Dasberg and Dalton (1985), Dalton and Van Genuchten (1986), Ledieu et al. (1986), Dalton (1990), Mualem and Friedman (1991), Noborio et al. (1994), and Heimovaara et al. (1995).

2.4.4.6 Frequency domain analysis of TDR waveforms

Coaxial cable testing equipment (TDR units) are rugged and battery-powered, and are therefore, practical instruments of field use. However, network analyzers have superior frequency domain capability. Although interpretation of measurements made by either system is based on the same principles, the fundamental difference between a TDR unit and a network analyzer is that the former measures the reflected signal as a function of time, while the latter measures as a function of frequency (Heimovaara et al., 1996).

Due to the fact that the frequency range generated by the signal from a TDR unit is wide and not well specified, it is difficult to compare TDR data with frequency domain data (Heimovaara, 1994). In order to compare these methods, a time domain deconvolution (TDD) method has been developed which enables time domain waveforms to be related to the frequency domain utilizing a Fourier transformation. This technique permits the frequency domain characterization of dielectrics, thereby enabling the complex dielectric constant (K^*) to be determined utilizing TDR.

Similarly, a network analyzer can be utilized to generate a TDR waveform (Heimovaara, 1996). A network analyzer is configured to sample the reflected signals from multiple equidistant frequencies (approximately 500) ranging from 300 kHz to 3 GHz. The data is transformed to the time domain by utilizing an inverse Fourier transform.

Further experimentation utilizing time domain deconvolution (TDD) can be found in Artacho et al. (1995) and Reader et al. (1995).

2.5 Methodology selection based on preliminary literature review

From a preliminary literature review of hydrogen nuclei detection (neutron probes), frequency domain analysis, and time domain analysis, it was necessary to determine

which technique would be most suitable for determining the water content of mine tailings. Other factors which were taken into considerations were: time constraints of a Master of Science thesis, research budget, and equipment availability.

Gringof and Nabiev (1987) published a list of criteria for selecting a method for determining moisture content:

- ⇒ selectivity criteria, which is determined by the dependence of the measured parameter on soil moisture content;
- ⇒ destructivity criterion, which is relative to the degree of destruction which the soil undergoes;
- ⇒ accuracy criterion, which is determined by the consumer's requirement for the measuring device;
- ⇒ continuity criterion, which is determined by the possibility of always measuring soil wetness at the same point of the soil profile by a continuous method;
- ⇒ inertia criteria, which is determined by the reaction time of the measurement device to a sharp change in wetness;
- ⇒ distribution criterion, which is determined by the possibility of variation of wetness in the soil profile in layers (with a 10 cm gradient) to a depth of 150 cm;
- ⇒ signal form criterion, which is determined by the technical resolution of the transformation of the nonelectrical magnitude of wetness into an electrical magnitude;
- ⇒ safety criterion, which is determined by the technique of labor safety and conservation.

These criteria present a helpful set of guidelines for selecting an appropriate moisture content measurement technology.

Four issues are immediately relevant to the use of neutron probes in mine tailings. The first is the issue of safety with regards to the use and training of personnel to operate a probe with a radioactive source. The second issue is related to field use of neutron probes. It is necessary to install an access tube prior to measuring soil moisture content. This procedure may be difficult and costly in many situations. The third issue is related to laboratory calibrations. According to Silvesti et al. (1991), the sample size required to calibrate a neutron probe is approximately 100 litres. It would require several hundred litres of mature fine tailings (MFT) shipped with an approximate solids content of 33% to produce 100 litres of MFT at 60% solids. The fourth issue is related to the range of water contents over which neutron probes produce accurate measurements. Schofield et al. (1994) state that the neutron probe readings appear to attenuate as the volumetric water content approaches 40%. Figure 2.2 also brings into question the accuracy of the neutron method in soil with high volumetric water contents.

Use of the capacitance probes (frequency domain) was also considered. Tomer and Anderson (1995) reported similar degrees of accuracy between TDR and capacitance techniques. However, currently available commercial units require the installation of an access tube similar to the neutron probe.

The use of time domain reflectometry was selected for this project due to the apparent insensitivity of TDR measurements of volumetric water content to temperature, soil density, mineralogy, and salt content (Topp et al., 1980). TDR analysis also appeared simpler than frequency domain analysis. Rugged, commercially manufactured TDR equipment is readily available. TDR probes were also commercially available and appeared to be reasonably simple and inexpensive.

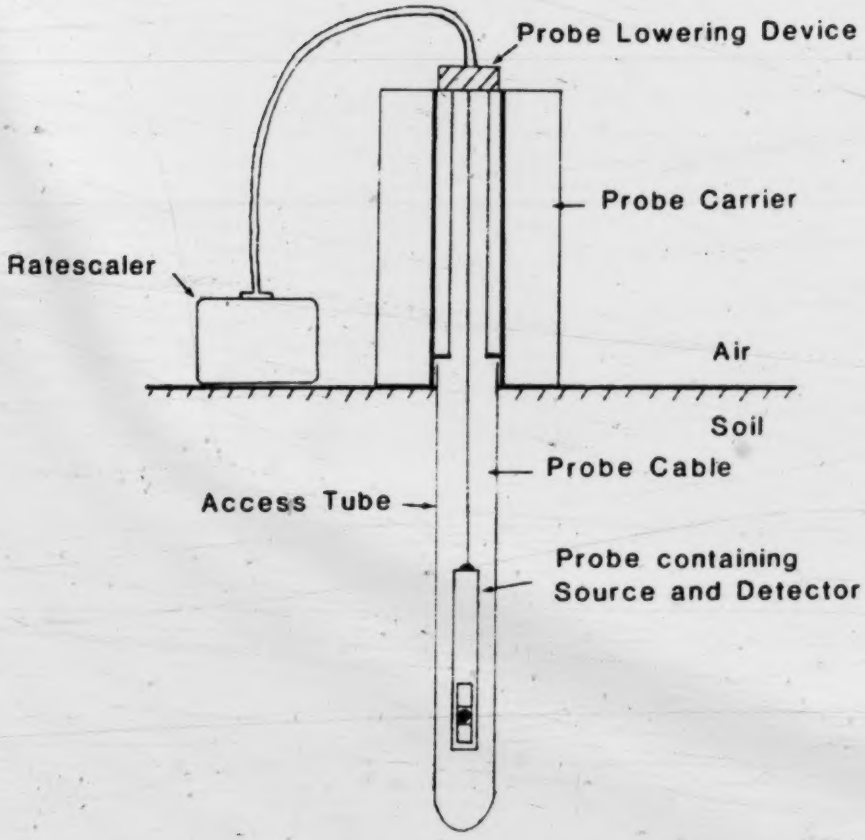


Figure 2.1 Principal components of the neutron probe (modified from Smith and Mullins, 1991).

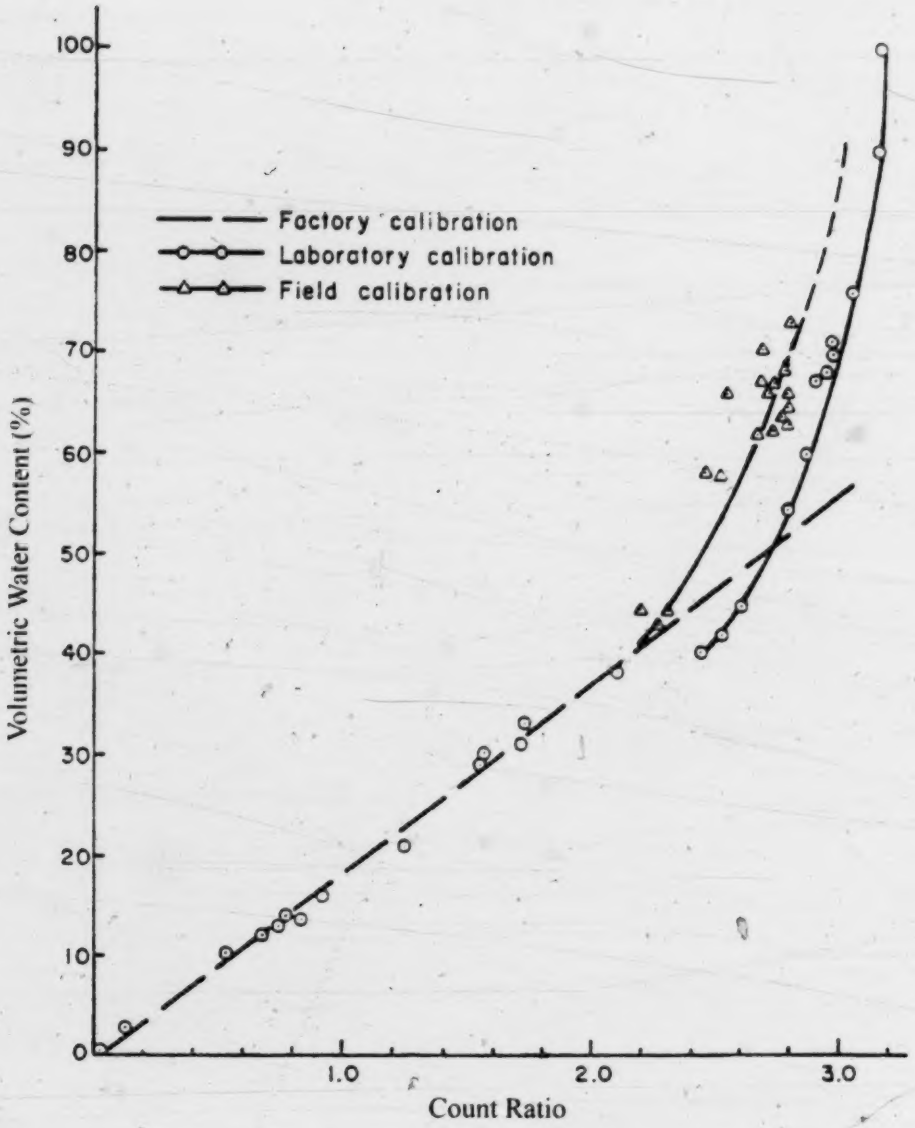


Figure 2.2 Neutron probe calibration curve (modified from Silvestri et al., 1991).

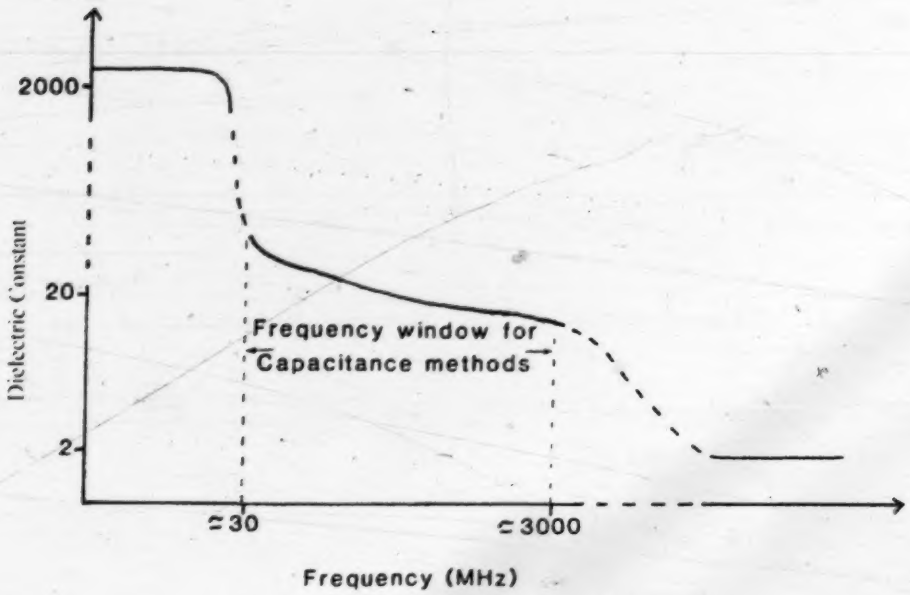


Figure 2.3 Dielectric constant versus frequency (modified from Smith and Mullins, 1991).

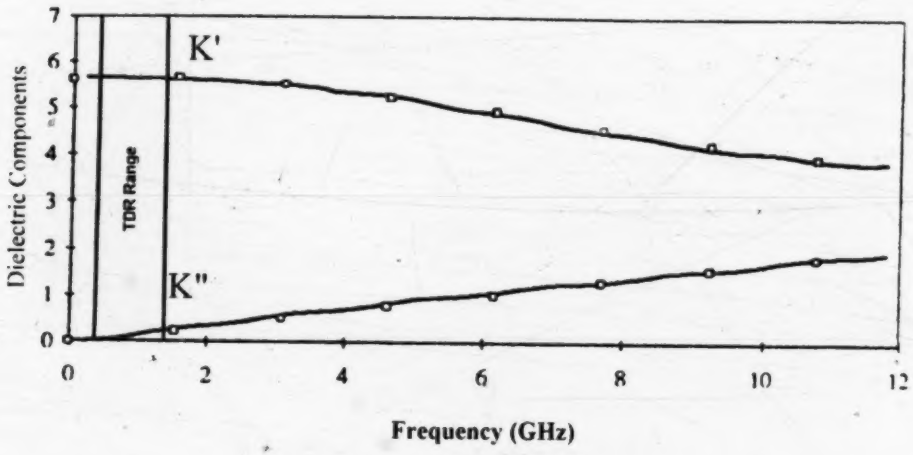


Figure 2.4 Components of the complex dielectric constant versus frequency for chlorobenzene at 24 °C (modified from Artacho et al., 1995).

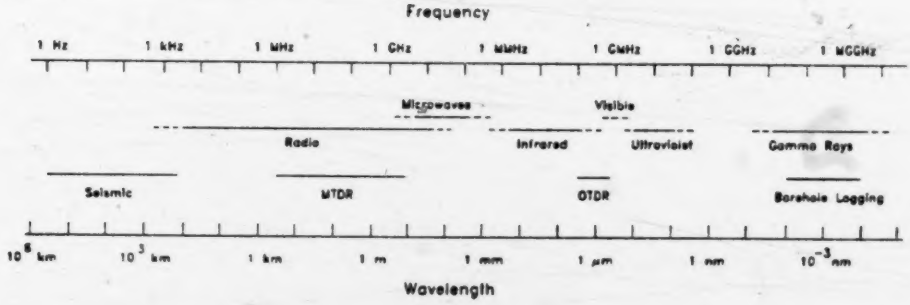


Figure 2.5 Electromagnetic spectrum (modified from O'Cónnor et al., 1994).

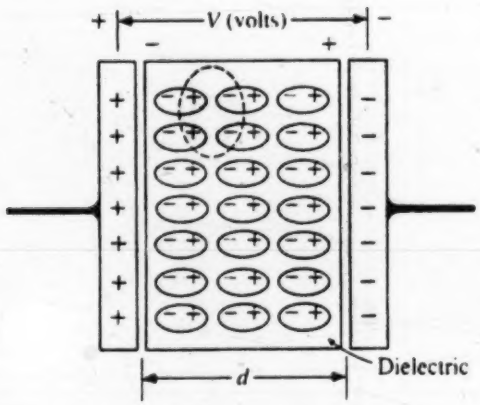


Figure 2.6 Dielectric material within a capacitor (modified from Boylestead, 1990).

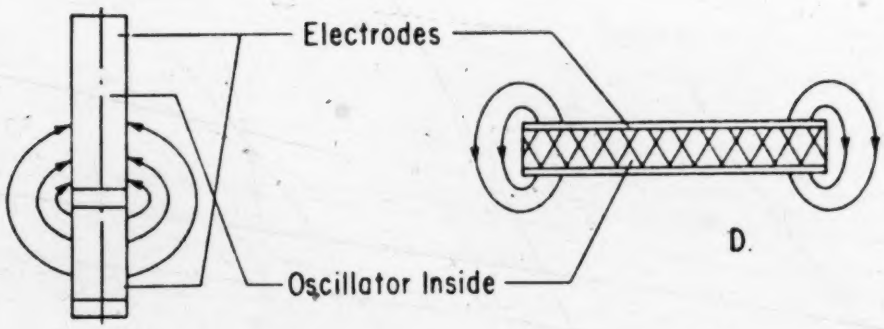


Figure 2.7 Capacitance probe electrode geometries (modified from Wobschall, 1978).

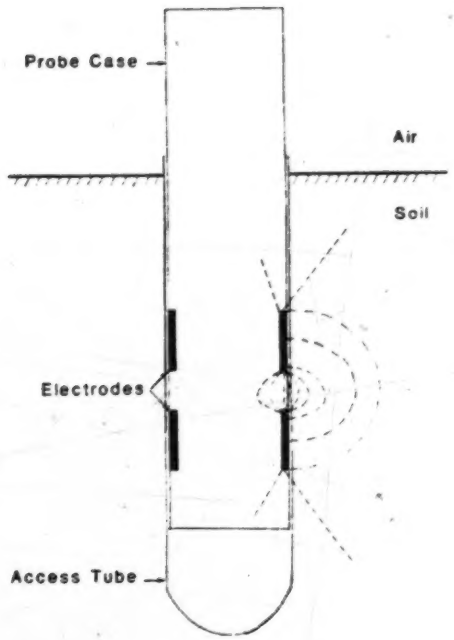


Figure 2.8 Capacitance probe electrodes installed within an access tube (modified from Smith and Mullins, 1991).

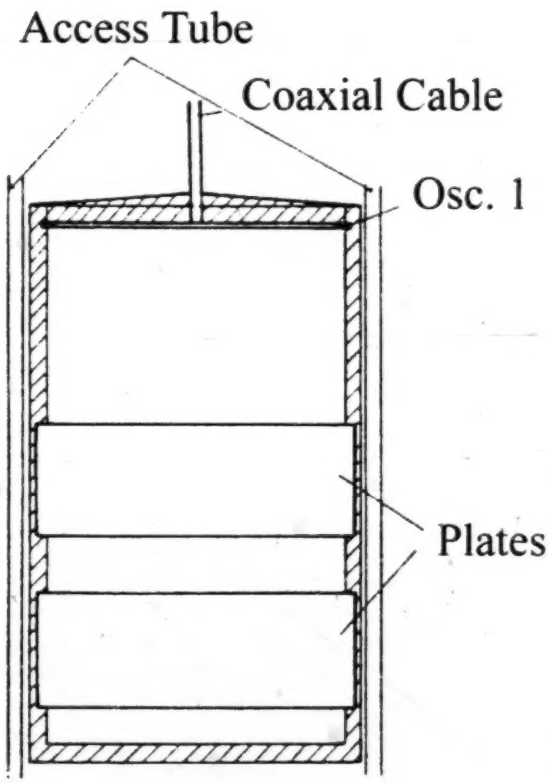


Figure 2.9 Capacitance probe schematic diagram (modified from Kuraz, 1981).

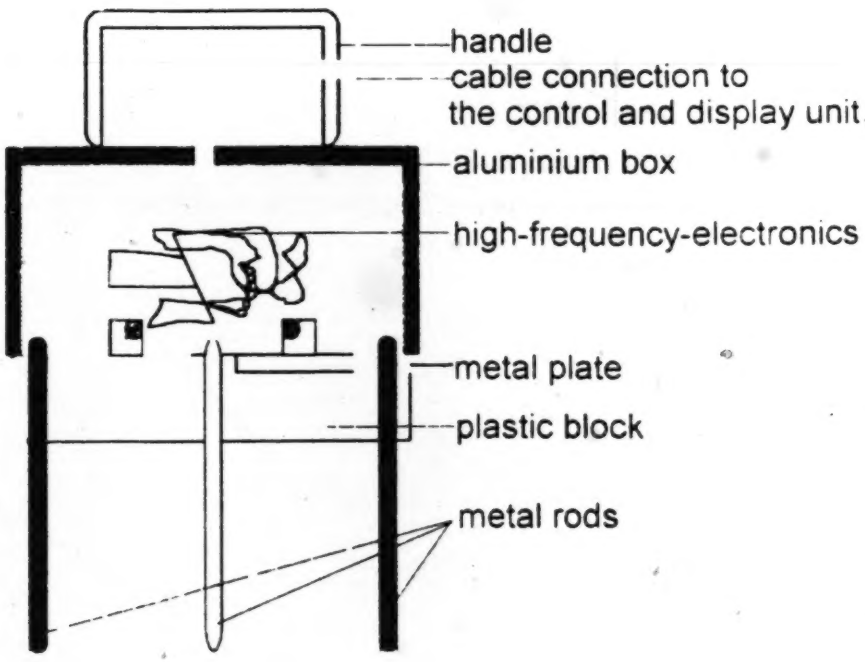


Figure 2.10 Portable capacitive probe (modified from Eller and Denoth, 1996).

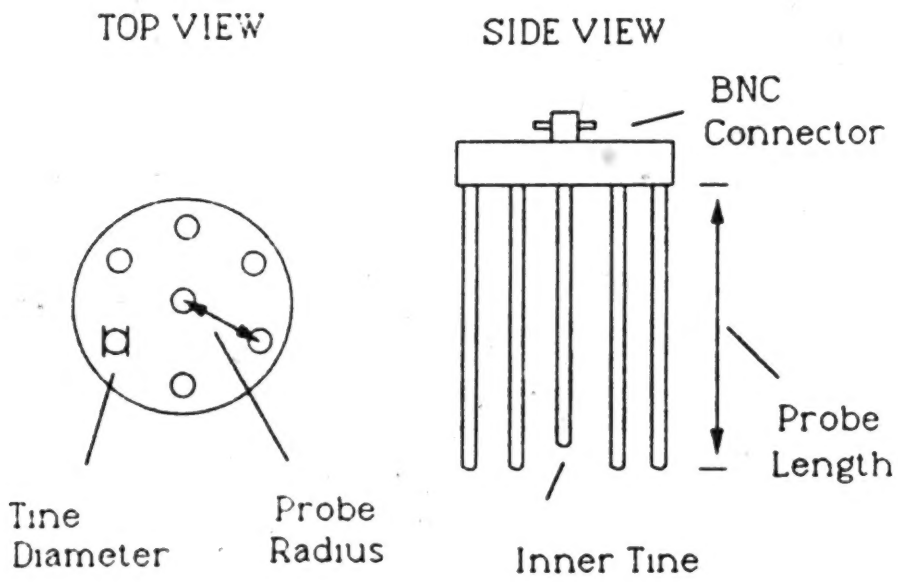


Figure 2.11 Impedance probe geometry (modified from Campbell, 1990).

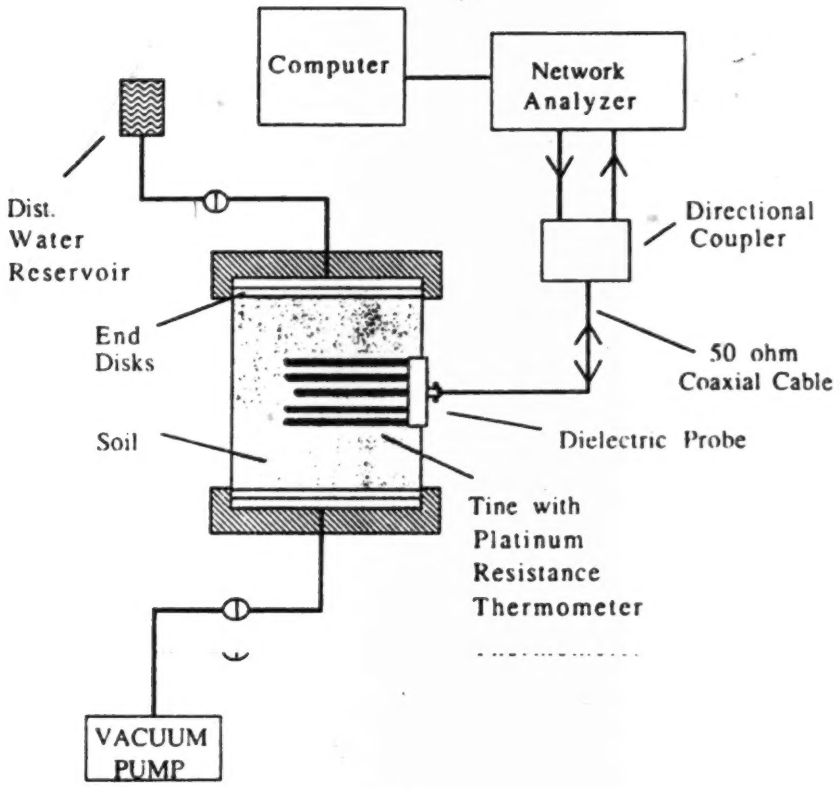


Figure 2.12 Impedance probe setup (modified from Campbell, 1990).

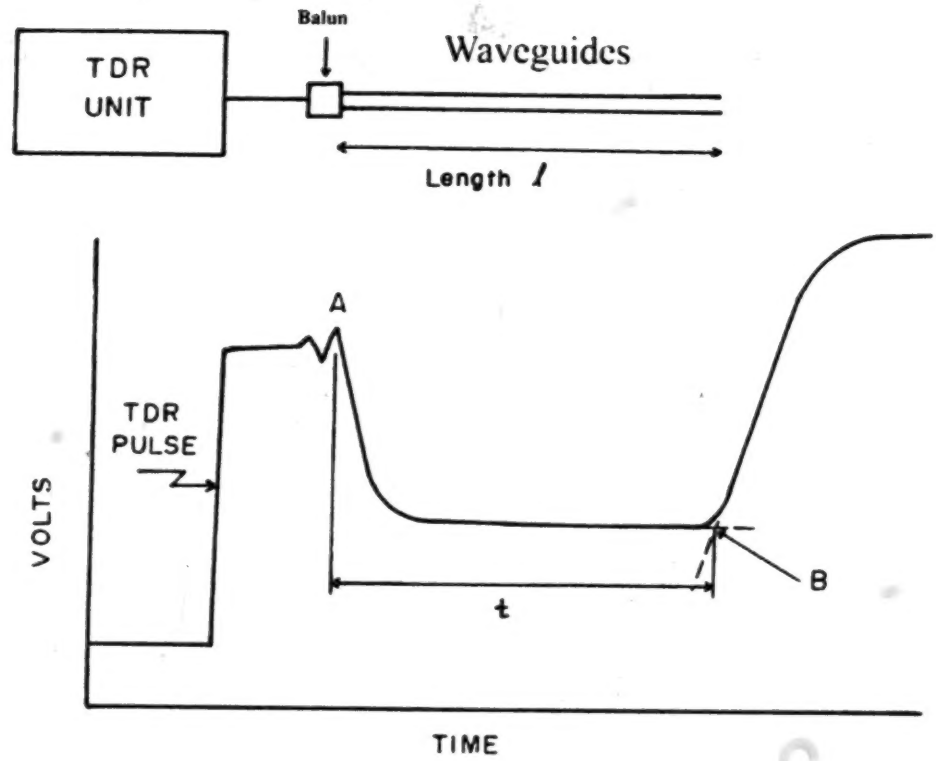


Figure 2.13 TDR components for measurement of soil moisture (modified from Topp and Davis, 1985b).

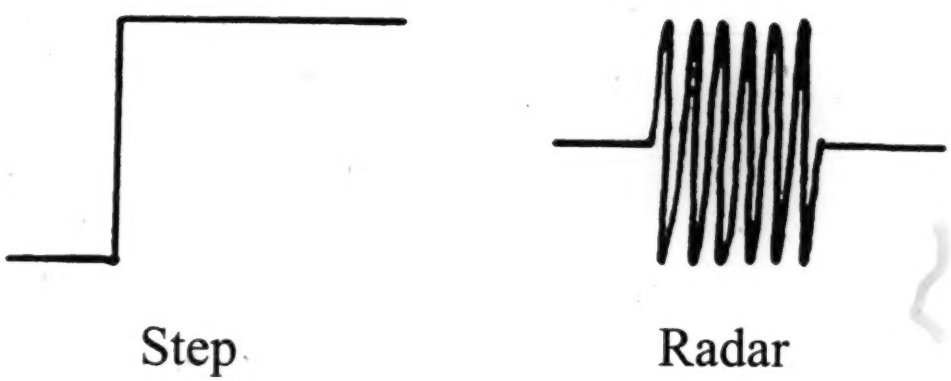


Figure 2.14 Common TDR pulses (modified from Andrews, 1994).

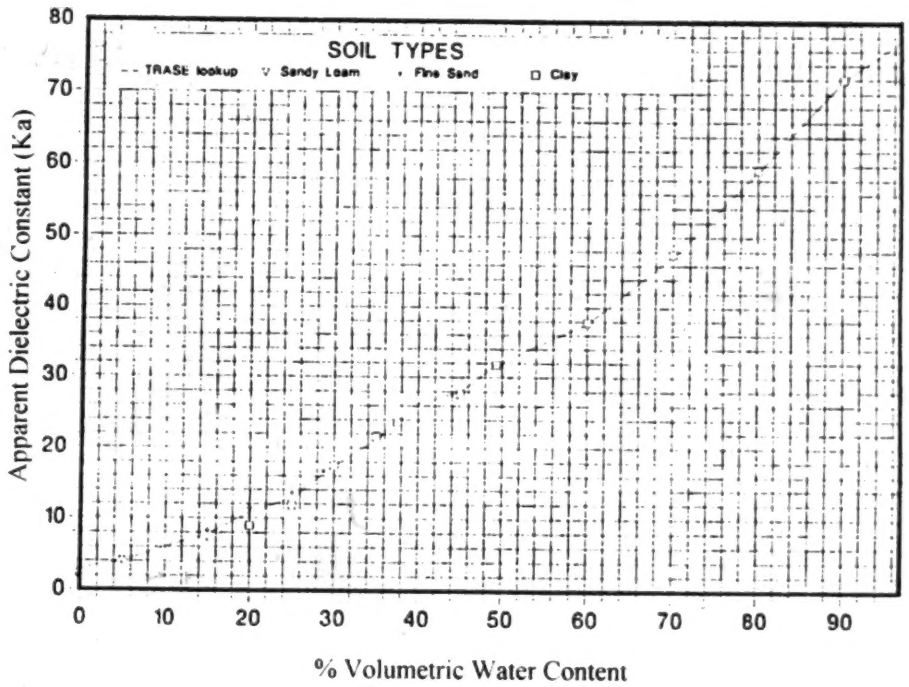


Figure 2.15 TRASE soil moisture table (modified from Skaling, 1992).

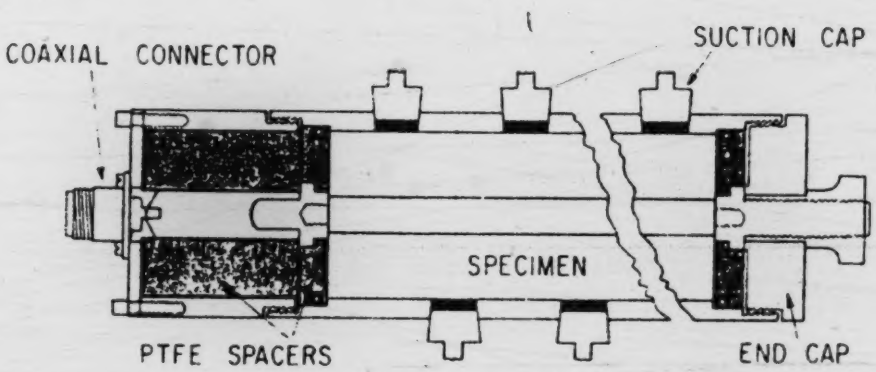


Figure 2.16 Coaxial soil container (modified from Topp et al., 1980).

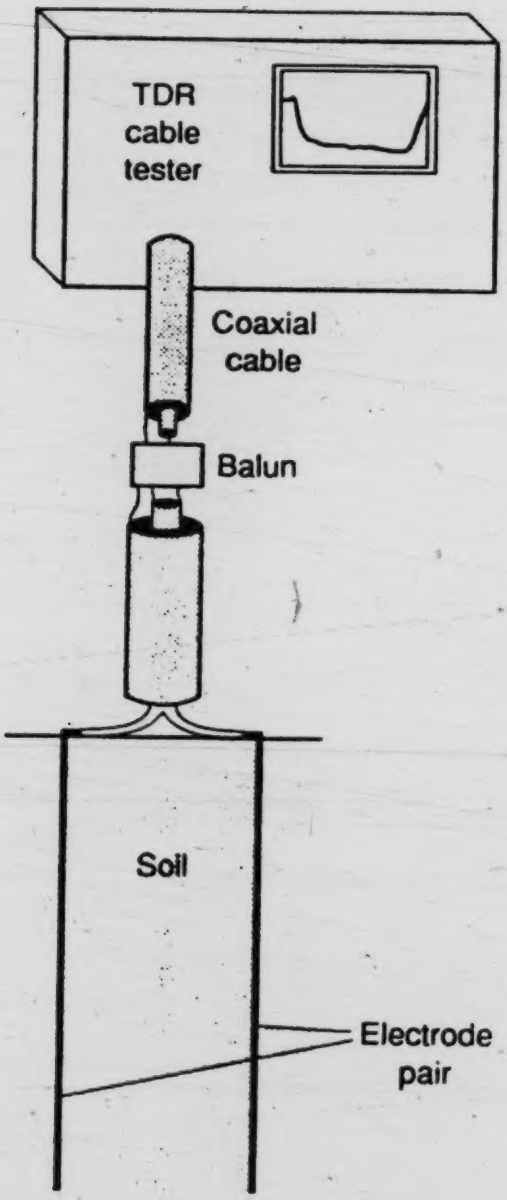


Figure 2.17 TDR circuit (modified from Herkelrath et al., 1991).

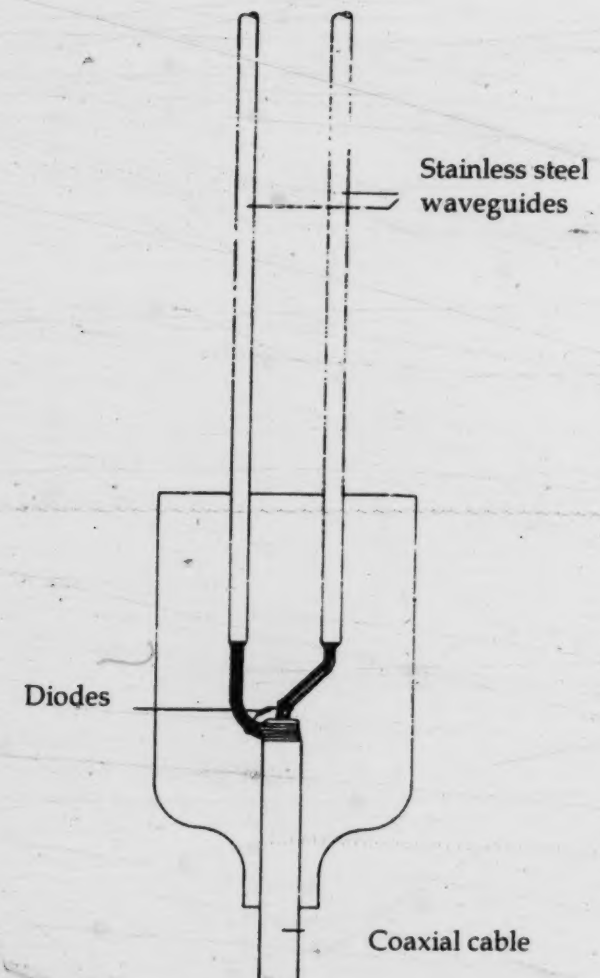


Figure 2.18 Parallel TDR waveguide design with diodes (modified from Ledieu et al., 1986).

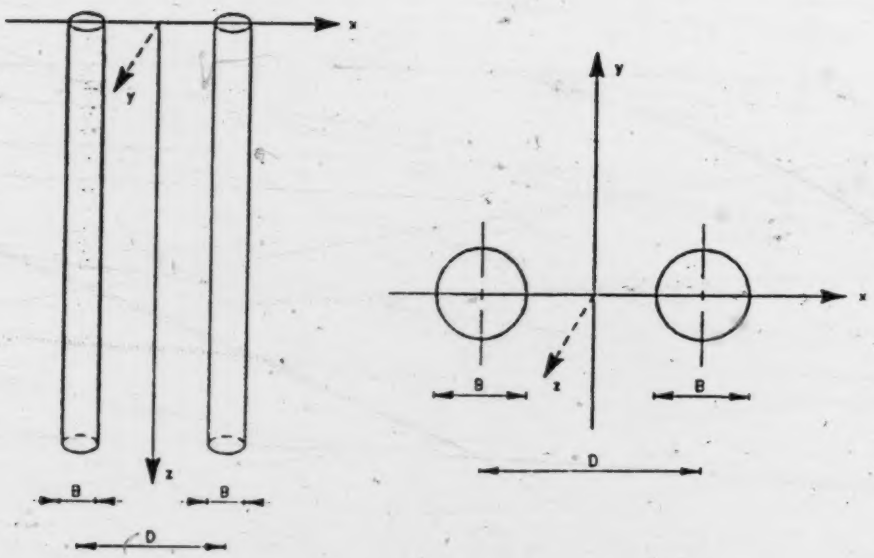


Figure 2.19 TDR probe configuration (modified from Petersen et al., 1995).

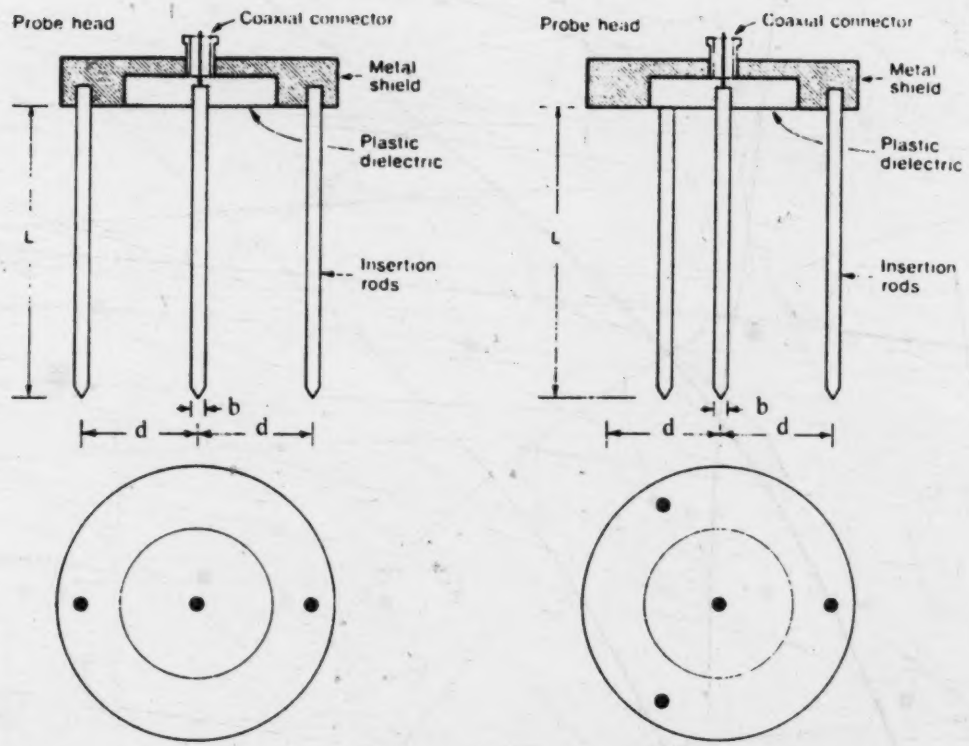
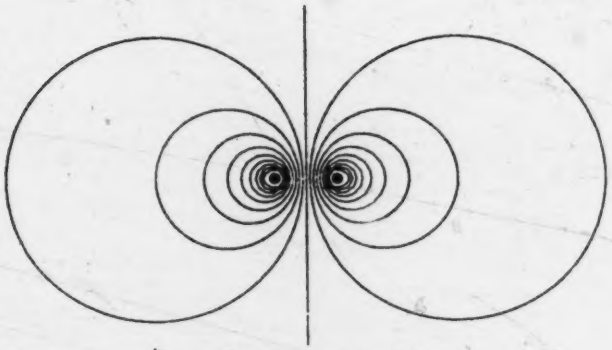
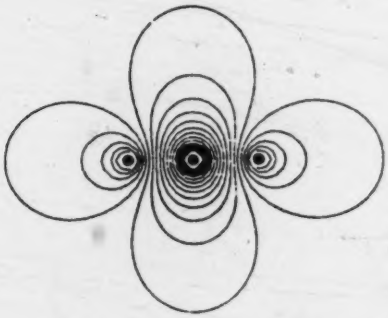


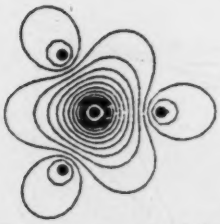
Figure 2.20 Three and four wire probe schematic diagram (modified from Zegelin et al., 1989).



2 - wire waveguide



3 - wire waveguide



4 - wire waveguide

Figure 2.21 TDR waveguide electric field distribution (modified from Zegelin et al., 1989).

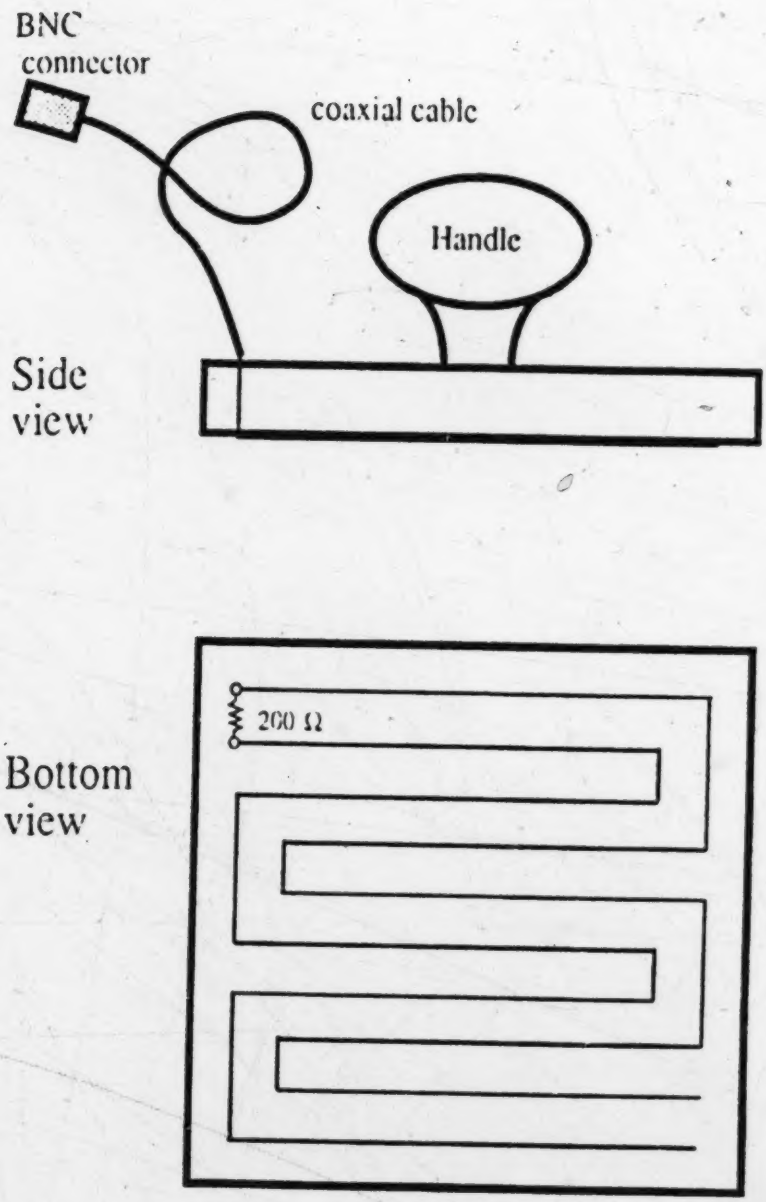


Figure 2.22 Serpentine parallel waveguides (modified from Selker et al., 1993).

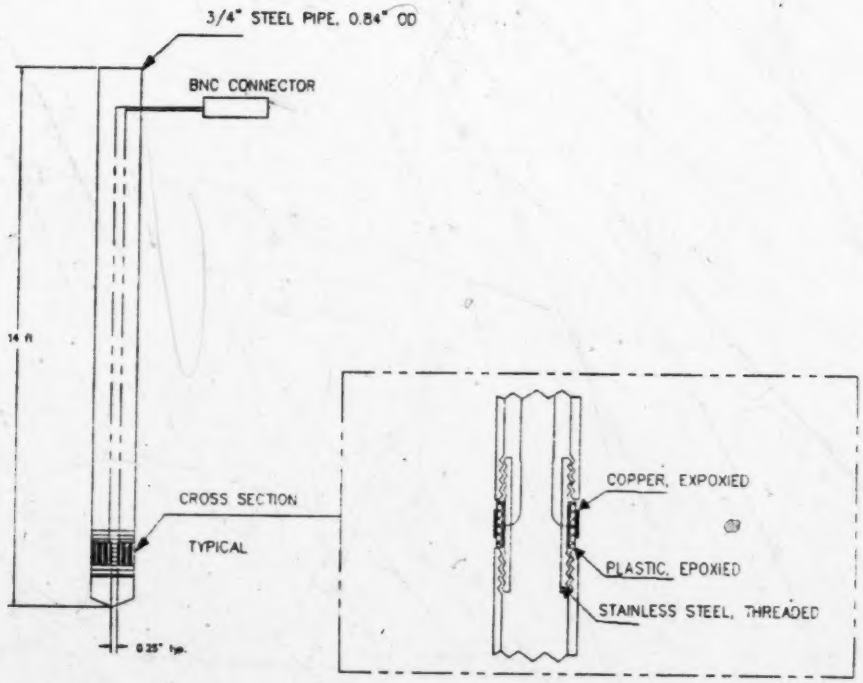


Figure 2.23 Insitu TDR probe (modified from Yokuda and Smith, 1993).

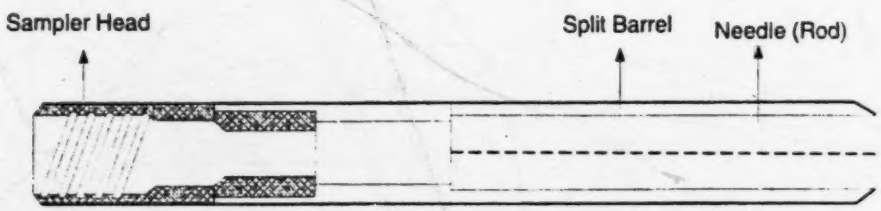


Figure 2.24 TDR split spoon sampler (modified from Kaya et al., 1994).

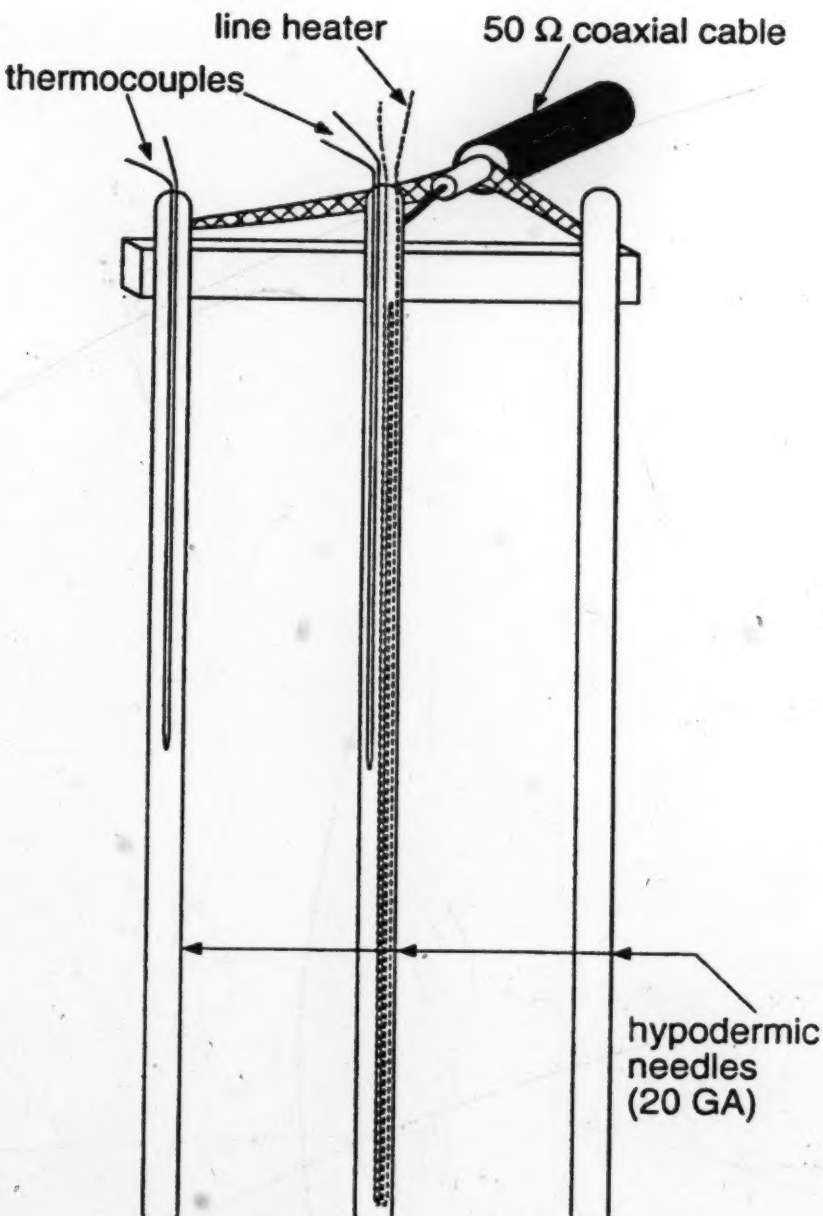


Figure 2.25 3-wire TDR probe for measuring soil water content, heat capacity, and thermal conductivity simultaneously (modified from Noborio et al., 1996).

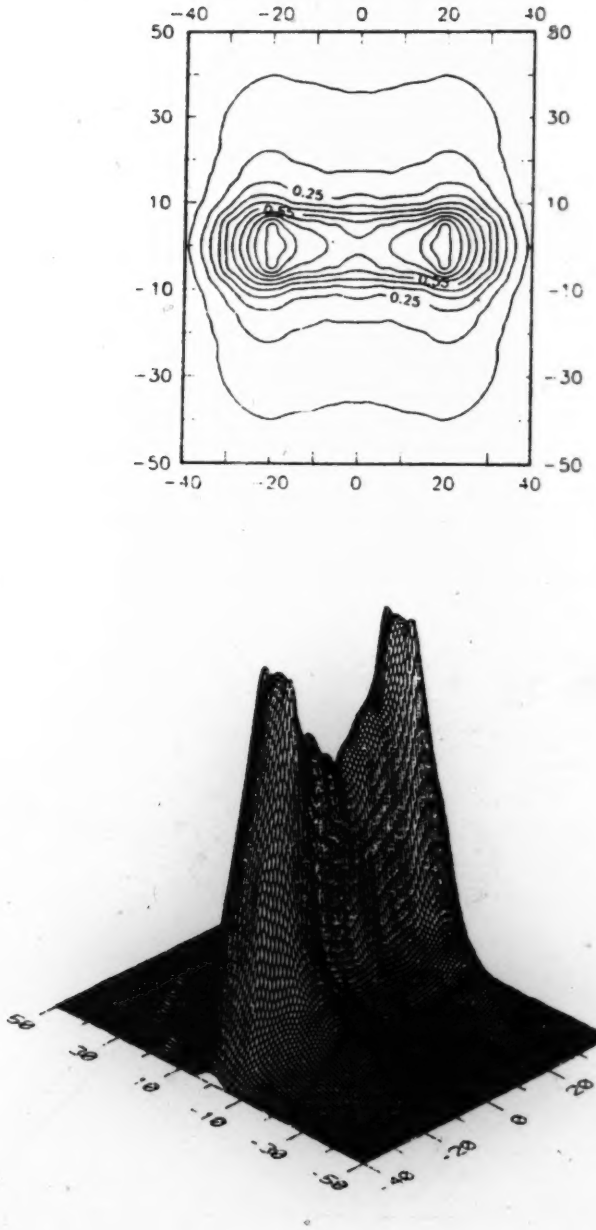


Figure 2.26 TDR probe spatial sensitivity (modified from Baker and Lascano, 1989).

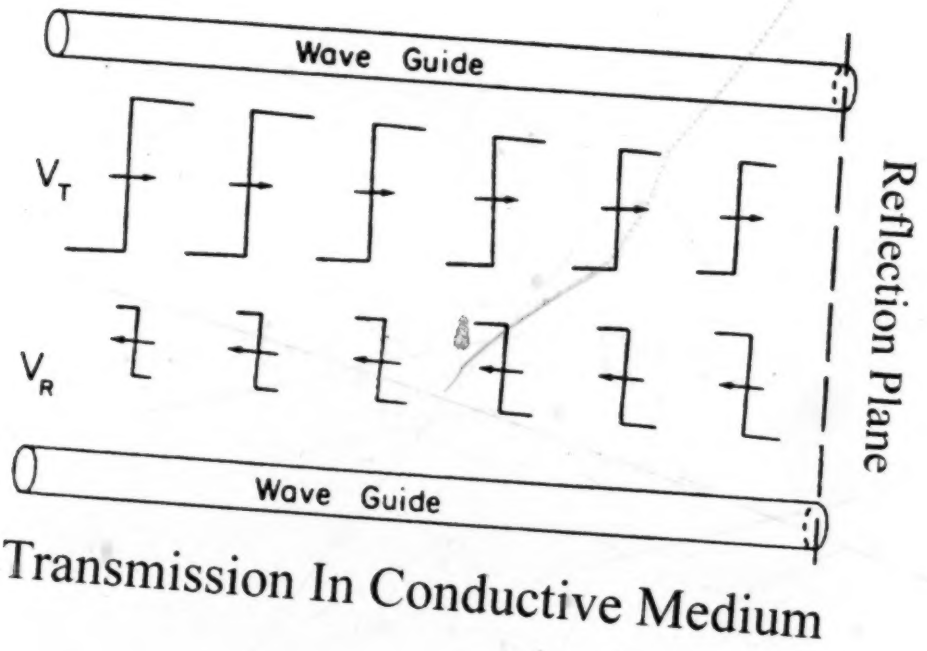


Figure 2.27 Schematic representation of transmitted and reflected voltage pulses in conducting media (modified from Dalton and Van Genuchten, 1986).

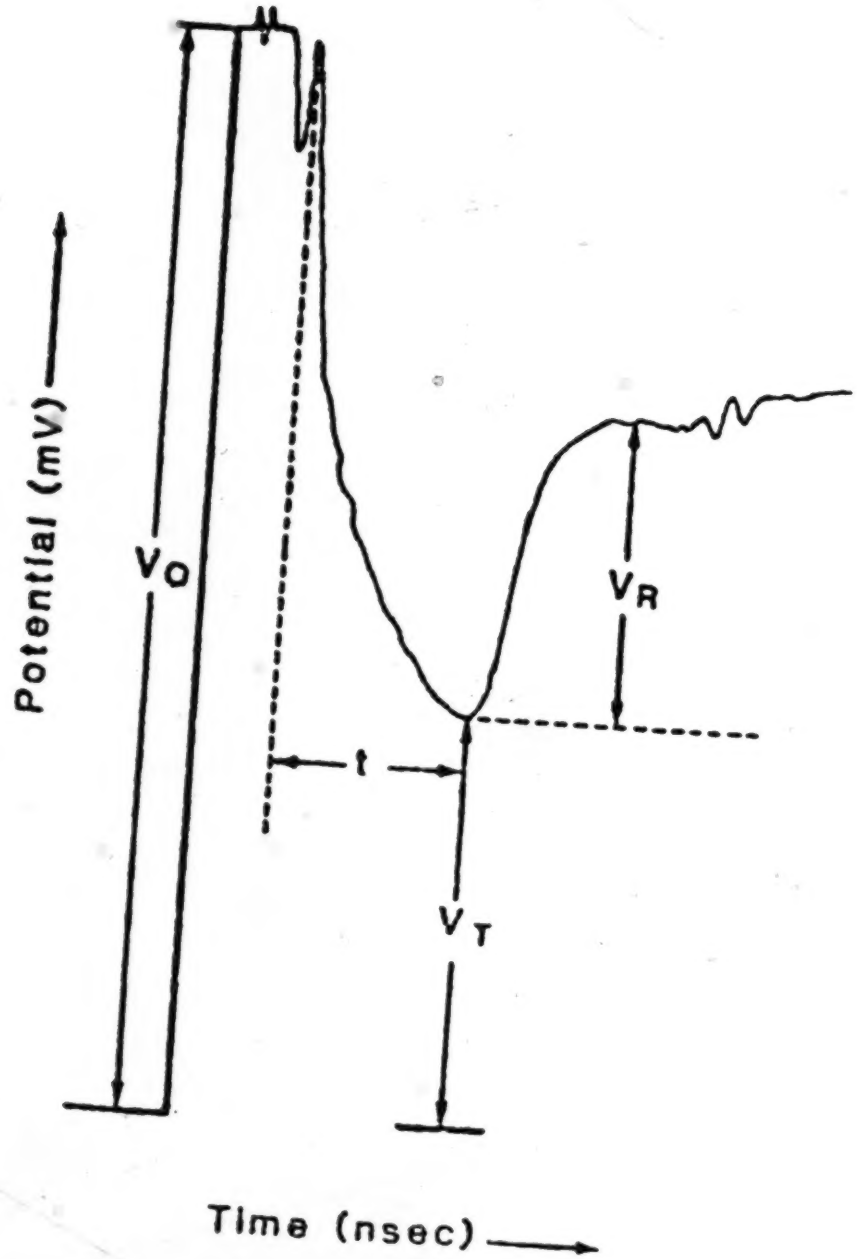


Figure 2.28 Typical TDR signal with important characteristics for calculating electrical conductivity identified (modified from Dalton et al., 1984).

Chapter 3

Equipment and TDR Waveform Analysis

3.1 Introduction

The initial objective of this project was to ascertain the feasibility of TDR for determining the moisture content of mine tailings and, if proven viable, to produce a field operable system for the characterization of Syncrude's mature fine tailings (MFT). Preliminary calibrations had been conducted at the University of Alberta utilizing a commercially available TDR unit produced by Soilmoisture Equipment Corporation. The intent of this thesis project was to acquire a similar commercial TDR moisture measuring device utilizing commercial probes / waveguides. Due to excessive delivery delays and the time constraints inherent with thesis deadlines, the acquisition of a Soilmoisture BE TDR unit was deemed impractical. Hence, other options were explored. This chapter briefly details the equipment utilized and related literature.

3.2 TDR units

Several manufacturers of TDR units are reviewed in the available literature (Malicki and Skierucha, 1989, Andrews, 1994, and Topp et al., 1996). These units are briefly summarized in Table 3.1, however, no critical evaluation of their performance was found in the literature. Topp et al. (1996) state that the Soilmoisture system requires the use of compatible probes. No further details are given, therefore, caution should be exercised when utilizing non-commercially manufactured probes with this system.

Based on the fact that most of the published literature regarding TDR calibrations for soil moisture content utilized either the Tektronix 1502, 1502B, or 1502C TDR unit, and with the apparent limitations of the Soilmoisture equipment stated by Topp et al. (1996), it was concluded that a 1502C made available by Syncrude would be utilized.

3.3 TDR probes

For preliminary experimentation, a Tektronix 1502C TDR unit was used in conjunction with Soilmoisture triple-wire waveguides / probes. Two types of waveguides were selected: 20 cm uncoated steel rod waveguides (model 6005L2) and 20 cm dielectric coated waveguides (model 600CL2). The selection of these probes was based solely on availability and the desire to utilize commercial equipment to assure the repeatability of analysis.

3.4 Coaxial cable

Several publications discuss the influence of cable length on the accuracy of TDR measurements. Herkelrath et al. (1991) state that long cables have a filtering effect on TDR signals. The cable attenuates the signal pulse making measurements with long cables impractical. Utilizing 50 cm long waveguides (d = 5 cm), the authors conclude that cables less than 27 meters in length have little effect on the calibration.

Heimovaara (1993) performed more extensive testing on the effects of cable length on 5 cm, 10 cm, and 20 cm waveguides. The author observed that coaxial cables, switches, and other connectors utilized for attaching TDR probes to the cable tester distorted the TDR waveform. Cables have a tendency to filter high frequencies, therefore reducing the frequency content from the TDR signal which results in a increase in the rise time of the pulse. The accuracy of dielectric measurements is diminished with long cable lengths due to a decrease in amplitude of the first reflection (start of probe) and second reflection (end of probe). The reflection amplitude can be optimized / steepened by utilizing smaller wire diameter (b) and shorter wire spacing (d). The authors conclude that the maximum practical cable lengths (utilizing RG 58 coaxial cable) are: 3 m for 5 cm waveguides, 15 m for 10 cm waveguides, 24 m for 20 cm waveguides.

The probes purchased from Soilmoisture were equipped with 2 meter cables. Two 10 meters extension cables were also purchased with the probes to investigate the effects of signal attenuation with total cable lengths of 2 m, 12 m, and 22 m in order to determine the maximum practical cable length for field use. For initial calibrations, the 2 m cable was utilized without extensions to minimize reflections at the connectors and signal attenuation.

The most common coaxial cable utilized for TDR measurements is 50Ω RG 58. This cable type is readily available and commonly utilized for computer network connections. However, lower signal losses may be attained by utilizing other cable types. This is further discussed in Zegelin et al. (1990) and Hook and Livingston (1995).

3.5 TDR waveform analysis

Figure 3.1 illustrates a typical waveform generated by a Soilmoisture uncoated triple-wire probe submersed in water. Early practitioners of the TDR method either photographed the TDR trace displayed on the cathode ray tube (CRT) or utilized a plotter and measured the signal trace manually. The methodology utilized in this manual technique to determine the start and end of probe is reviewed by Topp et al. (1982a) and Timlin and Pachepsky (1996). As illustrated in Figure 3.1, the travel time (t) is calculated graphically. Once the start and end of probe have been determined, equation 2.10 is utilized to calculate the apparent dielectric constant Ka. This technique for determining Ka has several disadvantages; graphical interpretation is slow, labor intensive and has a low degree of repeatability.

The manual calculation method has been gradually replaced with computer algorithms to find the initial and end points of the trace by locating the position of characteristic slope changes. Computer analysis methods are discussed by Zegelin et al. (1989), Baker and Allamaras (1990), Heimovaara and Bouten (1990), Zegelin et al. (1990), Herkelrath at al. (1991), Skaling (1992), Heimovaara (1993), Timlin

and Pachepsky (1996), and Topp et al. (1996). The computer algorithm commences by allowing the user to input the start point of the probe. The start point selection may also be automated by utilizing a shorting diode illustrated in Figure 2.18 or the methodology reviewed in Topp et al. (1996). However, the start point is most commonly selected by placing the probe in a solution of known dielectric constant and selecting the position on the waveform which produces an apparent dielectric constant approximately equal to the known value. The end of probe is calculated by determining two points (Figure 3.2): the point of maximum derivative and the local minimum. Once the maximum derivative is found, the computer algorithm searches for a local minimum at a distance approximately 0.8 times the length between the start point and the maximum derivative. Line A is draw tangent to the maximum derivative while line B is drawn through the local minimum parallel to the x-axis. The end of the probe is considered to occur at the point of intersection of line A and B.

Other TDR practitioners have adopted variations of the aforementioned computer algorithm. The most notable alteration involves the calculation of line B (Figure 3.2). Heimovaara and Bouten (1990) describe a computer algorithm which places line B on a slope calculated by a weighted average of a given number of points to the left of the local minimum (Figure 3.3). This modification has minimal effects on reducing the apparent dielectric constant calculated.

TDR waveform analysis must be altered for calculating the apparent waveguide length (La) in air (Figure 3.4). The computer algorithm calculates La as half the distance between the start of probe and the local maximum.

The waveform output from a Tektronix 1502B/C TDR unit illustrated in Figures 3.1 - 3.4 are in units of length (meters) along the x-axis. Hence the apparent dielectric constant can be calculated by the following equation, where L is the actual length of the probe, La is the apparent probe length calculated from the TDR

waveform and Vp is the velocity propagation constant of the TDR unit (Kaya et al., 1994):

$$Ka = (La / (Vp\ L))^2 \qquad (3.1)$$

The value of Vp is dependent on the TDR settings. The literature indicates that a value of Vp = 0.99 is commonly utilized. However, Ka is independent of Vp as La is a function of Vp, the speed of light and time:

$$La = (Vp)\ c\ t \qquad (3.2)$$

Substituting equation 3.2 into 3.1 results in equation 2.10 which is independent of Vp. Selecting a value of Vp based on the characteristics of the coaxial cable (Vp = 0.66 for RG 58 cable) is helpful for locating the TDR probe waveform when utilizing longer cable lengths (greater than 2 m).

3.6 Software availability

Several software packages are currently available for TDR waveform interpretation with Tektronix 1502B/C units. The computer algorithm described by Heimovaara and Bouten (1990) is available from the authors, and a Windows TDR software package entitled Win_TDR is available from the Utah State University soil physics group.

For the purposes of this project, in the interest of avoiding a "black box" solution from software written by third parties, a Windows software package for TDR waveform analysis was written based on the work of the authors listed in section 3.5. The intended use of this software package is solely the measurement of apparent dielectric constant with both comercially non-commercially manufactured probes.

3.7 M-TDR version 1.1 operating manual

M-TDR v1.1 was written to calculate the apparent dielectric constant (Ka) and refractive index (N) utilizing a Tektronix TDR unit. The waveform analysis algorithm permits the user to modify the parameters for determining the maximum first derivative and local minimum. This ensures the waveform analysis will be compatible with most waveguide geometries.

3.7.1 Hardware requirements

M-TDR is a 16 bit Windows application compatible with MS Windows 3.1 or higher. The software will operate on any 386 or later CPU with an available serial for communication with the TDR unit.

M-TDR can be utilized to operate any Tektronix 1502B/C TDR unit equipped with ROM version 5.0 or later, and a SP232 serial extended function module. The SP232 signal levels conform to EIA RS232-C specifications (Tektronix, 1989) with the following configuration:

AA (pin 1, protective)
AB (pin 7, signal ground)

BA (pin 2, TXD - asynchronous data transmitted from DTE to DCE)
BB (pin 3, RXD - asynchronous data received by DTE from DCE)
CA (pin 4, RTS - request to send signal originated by DTE)
CB (pin 5, CTS - clear to send signal originated by DCE)

CC (pin 6, DSR - data set ready).

3.7.2 Software Installation

The M-TDR software package includes 3 floppy disks. The program is installed in Windows by inserting disk #1 into the floppy drive and running a:\setup. The installation software will automatically install all required drivers and cue the user when to insert disks #2 and #3.

3.7.3 Software setup

Before M-TDR can be utilized to measure Ka and N, the software must be configured for the TDR unit and waveguides to be utilized. Figure 3.5 illustrates the M-TDR main screen. Three pull-down menus appear at the top left-hand corner of the screen: *File, Parameters*, and *Communication.*

3.7.3.1 Communication

When the Communication pull-down menu is clicked with the mouse, two sub-menu items appear: *Protocol* and *Resume TDR*. The RS232 serial cable communication parameters can be modified by clicking on the *Protocol* sub-menu. Figure 3.6 illustrates the pop-up *Protocol* menu. From this menu, the comport and baud rate can be set as required. The factory set baud rate for the SP232 serial extended function module is 1200. This rate may be altered by disassembling the SP232 and repositioning the jumpers on the circuit board (refer to section 8 of the service manual - Tektronix, 1989).

3.7.3.2 Probe

The probe input parameters can be modified by clicking on the *Probe* sub-menu located in the Parameters pull-down menu. From the *Probe* pop-up menu (Figure 3.7), the probe waveguide length (mm), cable length (m) and probe type (standard or diode) can be altered. The cable length can also be modified by dragging the horizontal scroll bar (HSB) located below the TDR Wave Output on the main screen. The selected cable length is outputted below the HSB as illustrated in Figure 3.5 for a 3.6 meter cable length (Distance = 2m).

3.7.3.3 TDR settings

The TDR unit input parameters can be modified from the *TDR Settings* pop-up menu (Figure 3.8) located in the *Parameters* menu. The velocity of propagation (Vp), distance per division (meters / div.) , vertical scale (Millirho), and noise filter can be set.

3.7.3.4 Wave analysis

The *Wave Analysis* pop-up menu (Figure 3.9), located in the *Parameters* pull-down menu, allows the parameters utilized for calculating the apparent length of the waveguides to be modified.

The Min. Start variable controls the point between the start of probe and maximum first derivative where the program will commence the search for the local minimum. For most probe gometries, the default value (0.8) is recommended.

The Lines Visible option button enables / disables the visibility of the lines which illustrating the local minimum and maximum first derivative utilized to calculate the end of the probe.

The Smooth Wave option button enables / disables a waveform smoothing function.

The Slope Smoothing Sensitivity variable controls the number of points utilized in the moving average algorithm to smooth the first derivative. Typical values for the variable range from 2 - 10. This variable effects the value of the maximum first derivative and should be optimized for each probe geometry.

The Base Slope Sensitivity variable controls the number of points to the left of the local minimum utilized to calculate slope. If the default value of zero is utilized than a line through the local minimum, parallel to the x-axis, is used to determine the end of the probe.

3.7.3.5 Input parameters display box

The input parameters can be reviewed in the Input Parameters display box located at the bottom left-hand corner of the man screen.

3.7.4 System initiation

Prior to initiating the system, ensure that the power for the TDR unit is on and the serial cable is properly connected between the 1502B/C and the PC. The TDR unit parameters discussed in section 3.7.3 are not sent to the Tektronix device until the *Initiate* command button is clicked. This button is located at the bottom right-hand corner of the main screen. Once the system initiation is complete the screen on the TDR unit should be off, the TDR unit controls locked, and the Data Output window should indicate "initiation complete". Manual control of the TDR unit can be released by selecting the *Resume TDR* command located in the *Communication* pull-down menu.

Input parameters which are controlled by the *Initiate* command include the TDR Settings (Figure 3.8), the horizontal scroll bar (HSB) and vertical scroll bar (VSB). These parameters may be altered at anytime, however, the chances will not be sent to the TDR unit until the *Initiate* command button is clicked.

3.7.5 Loading TDR waveform

Once the TDR initiation procedure is completed, the *Get Wave* command button, located at the bottom of the main screen, is unlocked. A waveform may then be loaded and displayed in the TDR Wave Output display. The number of waveforms sampled is a function of the TDR unit noise filter (1 - 128 waves) and the # *Wave Avg.* variable input box located below the Data Output display on the main screen. The noise filter variable (TDR Settings menu) controls the number of waves sampled by the TDR unit and then sent to the computer via serial cable. The # *Wave Avg.* variable controls the number of waveforms called from the TDR and then averaged by the computer algorithm. For most practical applications, The # *wave Avg.* variable can be left on the default value of 1 while a noise filter value of 4 to 64 is recommended.

3.7.6 Selecting start of probe

Two methods are available to select the start of probe: click in the desired point on the TDR Wave Output display or click the - or + command buttons on the main screen.

3.7.7 Wave analysis

Once a wave is downloaded from the TDR unit, the *Analyze* command button is clicked to determine the waveguide's apparent length (La) and calculate the apparent dielectric constant (Ka) and refractive index (N). The following equations are utilized:

$$Ka = (La / (Vp\ L))^2 \qquad (1)$$

$$N = La / (Vp\ L) \qquad (2)$$

The results of these calculations are outputted in the Data Output display box located on the main screen.

3.7.8 Saving data

The waveform data, input parameters, and calculated Ka and N can be saved by clicking the *Save* command button or selecting *Save Waveform* the File menu. The data is saved as a sequential file which can be retrieved utilizing spreadsheet software such as Excel.

Table 3.1 - TDR units.

TDR Manufacturer	Model number
Tektronix Inc.	1502 B or C
Soilmoisture Equip. Corp.	TRASE
Hewlett-Packard	54120B
HyperLabs	HL-1100

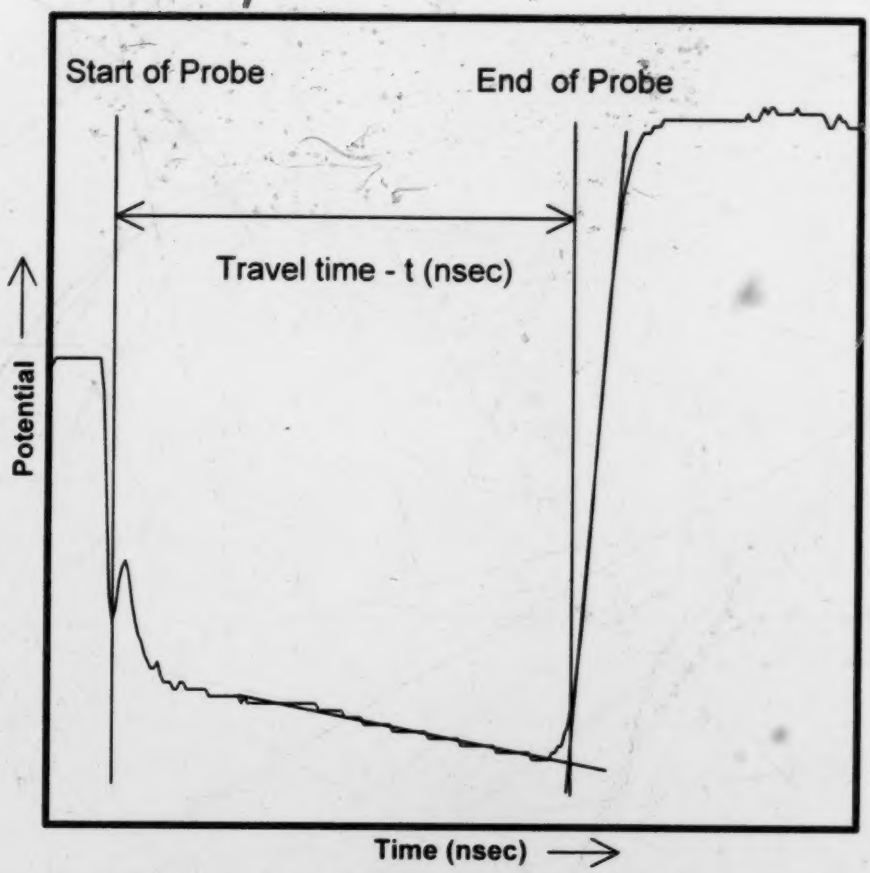


Figure 3.1 Manual analysis of TDR waveform.

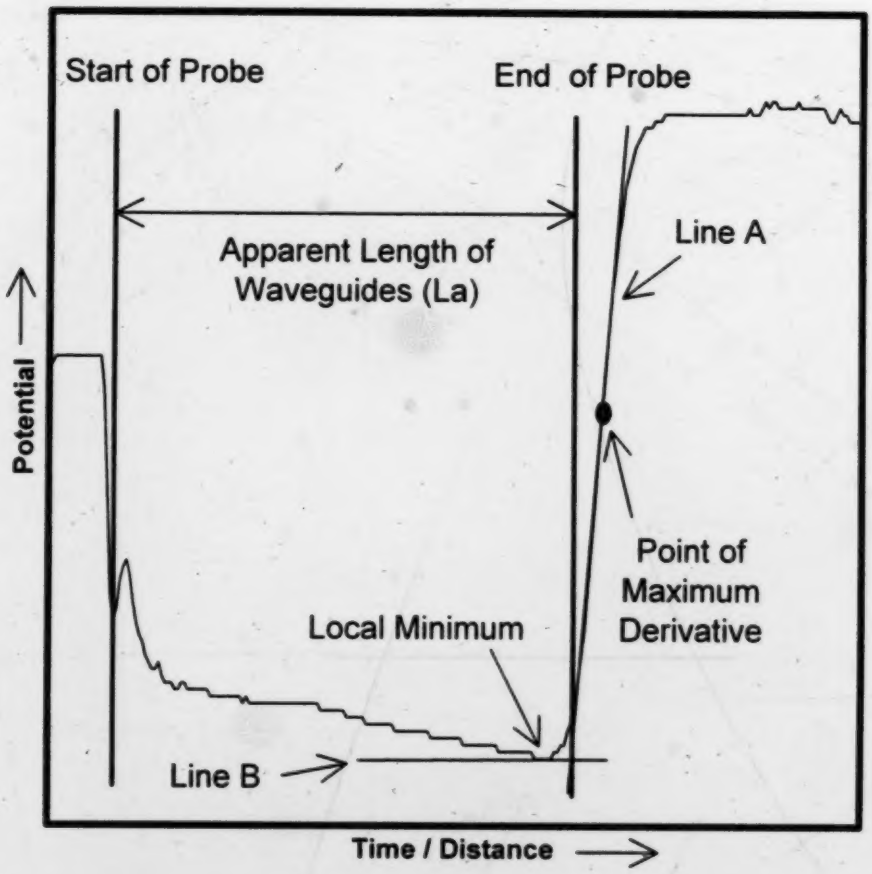


Figure 3.2 Computer algorithm for calculating La.

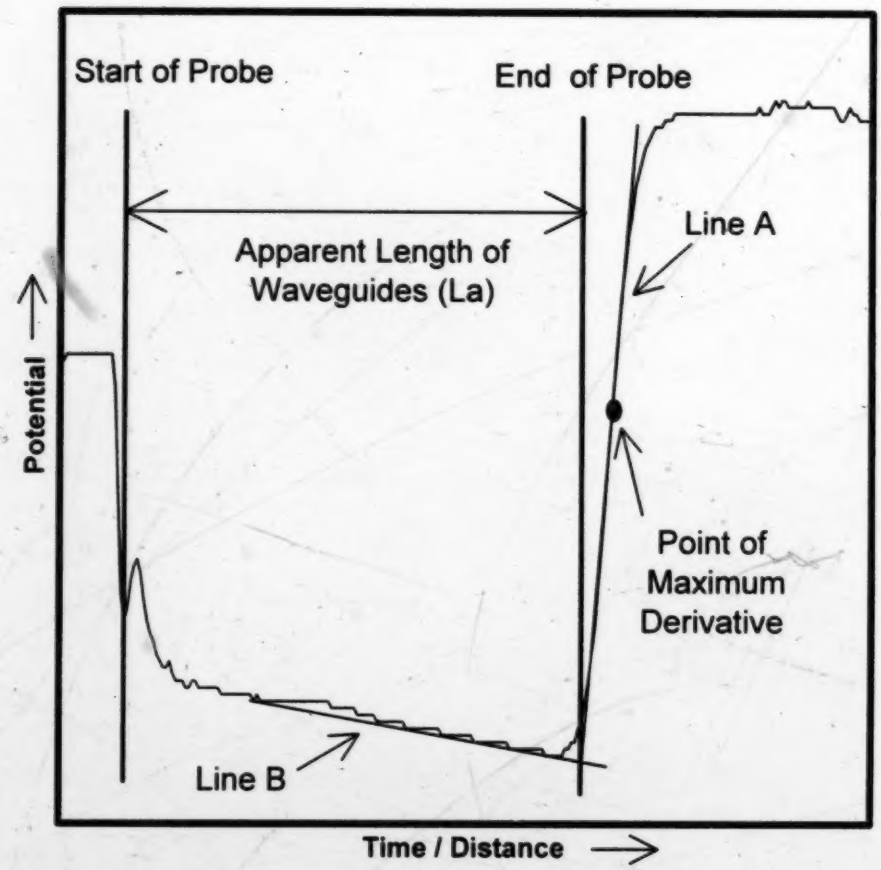


Figure 3.3 Computer algorithm for calculating La.

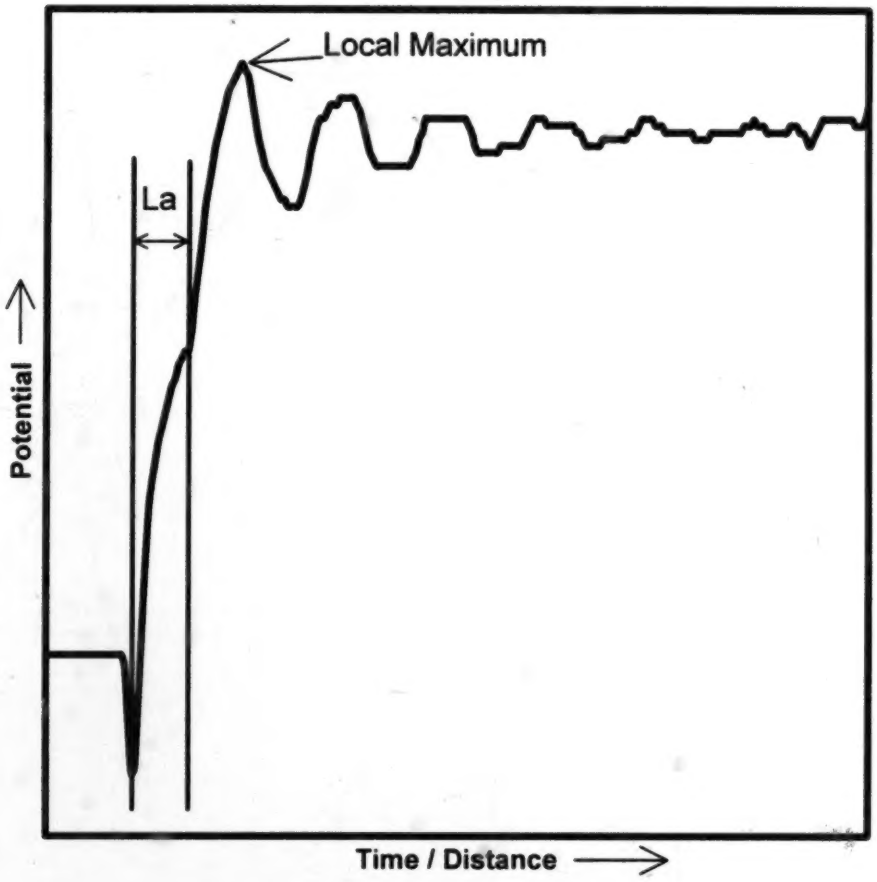


Figure 3.4 TDR waveform for probe in air.

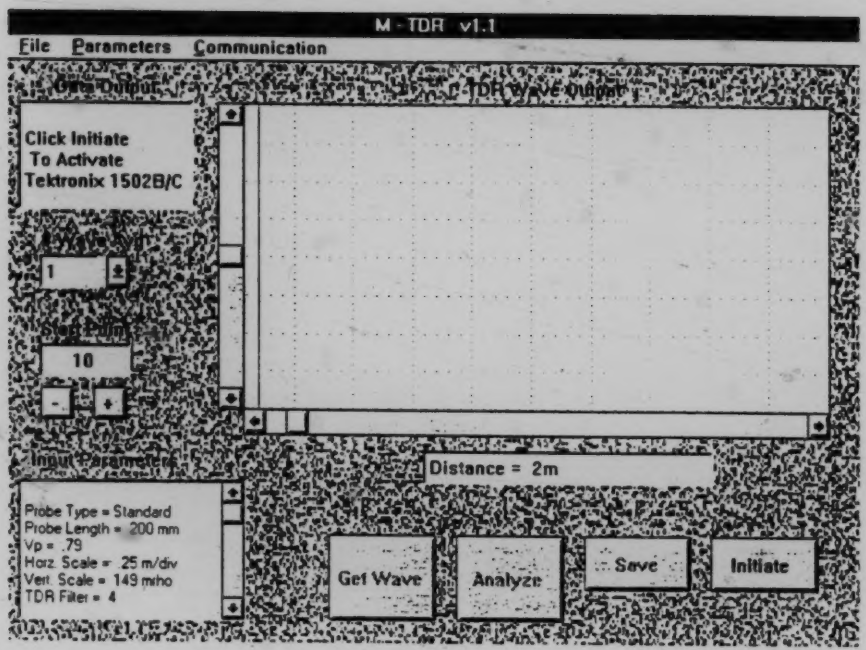


Figure 3.5 M-TDR main screen.

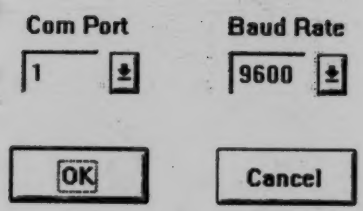


Figure 3.6 Communication protocol menu.

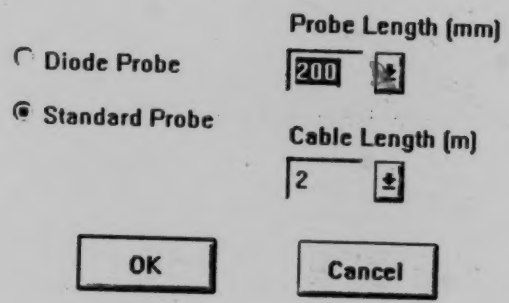


Figure 3.7 Probe settings menu.

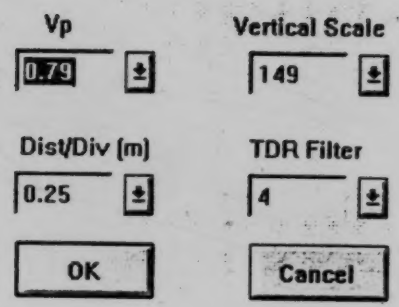


Figure 3.8 TDR settings menu.

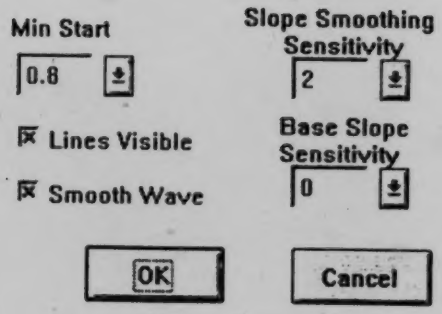


Figure 3.9 Wave analysis menu.

Chapter 4

Laboratory Procedure

4.1 Introduction

The laboratory procedure utilized for this experiment was primarily developed through experimentation. None of the literature regarding soil moisture content calibration methodology involved volumetric water contents greater than that found naturally in soils ($0 \leq \theta_v \leq 30\%$). Three media were tested for this project, distilled water, tailings pond water, and mature fine tailings. This chapter describes the equipment setup and calibration procedures utilized.

4.2 Equipment setup

The calibration procedure was conducted solely under laboratory conditions. The Tektronix 1502C TDR unit was connected to an IBM PC via RS-232 cable to download waveform data. Soilmoisture TDR probes were utilized with a 2 meter cable to minimize signal attenuation inherent with longer cables. Materials tested, including distilled water, pond water, and MFT were contained in 20 litre plastic containers (28 cm ∅ x 37 cm). This container was selected to ensure that the material being tested fully encompassed the zone of influence and the plastic container itself did not bias the results. The zone of influence, discussed in Baker and Lascano (1989) and Knight (1992), was estimated to encompass a maximum volume the length of the waveguides by a maximum radius 75 mm from the center wire (max. volume ≅ 3.5 litres). Therefore, placing the probe in the center of a 20 litre bucket ensured that the zone of influence would not be affected by the plastic bucket itself.

4.3 Material tested

The experimentation was divided into three sets of calibrations based upon the materials being tested: distilled water (to determine the accuracy of the probe over a

range of temperatures), tailings pond water / effluent, and mature fine tailings (MFT).

4.3.1 Distilled water

Calibrations were carried out on distilled water to determine the accuracy of the computer algorithm created to calculated the apparent dielectric constant. This was achieved by measuring Ka, for both uncoated and coated waveguides, over a temperature range of approximately 5 - 25°C and comparing the results with the known values calculated by equation 2.13. This temperature calibration was also carried out to determine the temperature sensitivity of the TDR technique for both waveguide types (uncoated and coated).

The effect of pH was also studied. The data published by the Fine Tailings Fundamentals Consortium, (FTFC, 1995) indicates the pH of mature fine tailings ranges from 7.6 - 8.4. Utilizing sodium hydroxide (NaOH) mixed in distilled water, the effect of pH was studied from approximately 7.0 - 11.0.

4.3.2 Tailings pond water

Calibrations were also performed on Syncrude tailings pond water to determine the effects of water chemistry on Ka relative to distilled water utilizing both uncoated and coated probes. It was necessary to compare the dielectric constant of distilled water versus pond water to determine if distilled water could be utilized for the calibration procedure (Ka versus volumetric water content) for the mature fine tailings (MFT). The effects of the pond water chemistry on the TDR signal were also studied to determine if greater signal attenuation in pond water would occur relative to distilled water.

4.3.3 Mature fine tailings (MFT)

Calibration curves for MFT were developed for a solids content range of 15 - 55%. Solids content is defined as (Dusseault and Scott, 1983):

Solids = mass of solids / total mass (4.1)

The MFT utilized in this experiment (barrel SY-24) was received with a solids content 33.8% (including bitumen). It was necessary to increase the solids content to approximately 55% in order to conduct the calibration procedure. Two methods were utilized to increase the solids content of the MFT: freeze / thaw consolidation and vacuum decanting. Two methods were selected to determine if the dewatering methodology would significantly effect the calibration results. The effluent from both dewatering methods was retained for use in the calibration procedure.

4.4 Probe calibration procedure

Prior to commencing TDR testing, it is necessary to calibrate the TDR unit and probe. This is achieved by manually determining the position of the "start of probe" line which will correctly calculate the dielectric constant of distilled water and air. These two media have dielectric constants which represent two extreme values (ε_{air} = 1 and $\varepsilon_w \cong 80$). The following calibration procedure was adopted for the Soilmoisture probes and the software developed for this project.

1. The probe is immersed in the center of a 20 litre bucket of distilled water of know temperature.
2. From equation 2.13, the dielectric constant of the distilled water is calculated.
3. The "start of probe" line is adjusted on the TDR waveform until the apparent dielectric constant calculated by the computer algorithm is approximately equal to the known value calculated in step 2.
4. The probe is removed from the water and dried. The dielectric constant is then check to assure that ε_{air} = 1.0.
5. If the dielectric constant of air is not measured by the computer algorithm as 1.0 then the "start of probe" line is adjusted.

6. The probe is submersed in the distilled water to check that the measured and theoretical dielectric constant values are still in agreement.
7. Repeat procedure until the measured values agree with the known values of ε_{air} and ε_w.

4.5 MFT calibration procedure

This tailings calibration procedure was utilized for all testing:

1. MFT (solids content ≥ 55%) is placed in a 5 gallon plastic bucket, to a depth exceeding 25 cm, to ensure the TDR probe is fully embedded and that the plastic material does not affect results.
2. The tailings are stirred with an electric mixer for two minutes to ensure the material is homogeneous.
3. The TDR probe is placed in the center of the tailings and five readings of dielectric constant are taken.
4. A sample (mass ≅ 200 g) is extracted from the center of the bucket, weighted, and placed in an oven at 105°C for thermogravimetric determination of water content.
5. 100 ml of effluent is added to the tailings.
6. Steps 2 -5 are repeated until a solids content of approximately 15% is attained.

The following assumptions are implicit with this calibration procedure:

⇒ the tailings mixture is homogeneous and the sample removed for thermogravimetric analysis is representative of the entire sample mass;

⇒ the gas generated by MFT is mixed out of the sample, therefore, a three phase mixture exists - mineral solids, effluent, and bitumen.

⇒ the bitumen content is relatively small and insignificant (approximately 1.8% by mass).

⇒ the tailings are 100% saturated.

The first assumption regarding homogeneity is readily justified by the mixing process. The second assumption is based on the observed bubbles which surfaced when the tailing were first mixed in the barrel in which they were received from Syncrude. After initial mixing, prior to dewatering, no gas bubbles were observed. Each sample was mixed prior to commencing calibrations and after each 100 ml increment of effluent is added. Therefore, it is assumed that any gas remaining is insignificant.

Bitumen is a complex mixture of organic compounds. The dielectric constant of complex hydrocarbon chains (C_nH_{2n}) is approximately 2.1 (Handbook of Chemistry, 1995). Therefore the apparent dielectric constant of bitumen is estimated between 2 and 3. Hence, the small percentage of bitumen in the mature fine tailings behaves dielectrically similarly to soil and does not significantly affect results.

The final assumption of 100% saturated conditions existing (zero air voids) is reasonable for the range of solids contents being measured. At 55% solids, the MFT is still in a gelatinous state and when thoroughly mixed, it is assumed no air voids are present.

4.6 MFT dewatering methodology

Two techniques were utilized to increase the solids content of the MFT which was shipped with a solids content of 33.8%: Freeze / thaw dewatering and vacuum decanting.

The freeze / thaw technique involves placing a 5 gallon bucket, filled with MFT, into a freezer unit for 48 hours. The bucket is then removed from the freezer and left undisturbed to thaw at room temperature. The effluent at the surface, is siphoned off and placed in a sealed container for use in the MFT calibration process. The remaining MFT is mixed and the cycle is repeated until a solids

content greater than 55% is achieved. Typically, 3 freeze / thaw cycles were required to increased the solids content to the desired value.

The vacuum decant method involved placing a vacuum filter tube (filter screen size = 0.22 micron) into an MFT sample and allowing the pore water to be sucked out of the MFT. This process was considerably slower than freeze / thaw dewatering and required approximately 10 weeks to reach 50 % solids. The effluent collected in the filter tube was placed in a sealed container for use in the MFT calibration procedure.

Chapter 5

Results

5.1 Introduction

This chapter summarizes the results of this project. The data analysis is divided into three sections: chemical and mineral analysis of the materials tested, effects of temperature, pH and chemistry on dielectric measurements, and MFT calibrations.

5.2 Chemical and mineralogical analysis

The following analyses were performed on barrel SY-24 which was utilized for all MFT calibrations performed during the course of this project. Grain size analysis, and liquid and plastic limits testing were conducted at the University of Alberta. Chemical analysis was performed at Norwest Labs.

5.2.1 Mature fine tailings

The results of the grain size analysis (hydrometer test) is presented in Figure 5.1. A typical grain size distribution range for MFT is presented in Figure 5.2. Comparison of these figures indicates that the MFT utilized for this project falls within the typical grain size distribution.

Liquid and plastic limits were also evaluated. The liquid limit was determined to be 47.0 % and the plastic limit was calculated to be 22.0 %. Typical liquid limit values for MFT range from 40 % to 75 % while plastic limit values range from 10 % to 20 % (FTFC, 1995).

A specific gravity (Gs) of 2.44 was also determined for the MFT. This value is slightly lower than typical values of 2.55.

A soil analysis was also performed to determine pH, electrical conductivity, soluble salts concentration, and water content. The results of this analysis are presented in

Table 5.1. The chemistry of MFT barrel SY-24 is similar to that published in FTFC (1995). However, there is more chloride and less sulphate than predicted by the aforementioned document.

5.2.2 Water analysis

Chemical analysis was performed on 1 litre samples of Syncrude tailings pond water, freeze / thaw effluent, and vacuum effluent. The results of this analysis is presented in Table 5.2.

The most significant findings from the water analysis report involved the pH, electrical conductivity, and sodium concentration. The pH level of the tailings pond water is significantly lower than the reported average values of 8.0 - 8.3 (FTFC, 1995). The electrical conductivity was also significantly higher than the typical values of 1250 - 2400 μS/m. Finally, the sodium concentration, which appears to be the dominant cation in Table 5.1, is significantly higher in the pond water than the expected concentrations 475 - 550 mg/L.

5.3 Distilled water and effluent calibrations

Initial calibrations were performed to determine the sensitivity of uncoated and coated TDR waveguides to variations in temperature. The results of these calibrations are shown in Figure 5.3. The apparent dielectric constants calculated with the uncoated probe is consistent with known values (equation 2.13). At a given temperature, the apparent dielectric constant calculated with the coated probe was significantly lower than that of the uncoated probe. This can be explained by the influence of the dielectric coating on the center rod which reduces the apparent length (La) of the TDR waveform as illustrated in Figure 5.4.

Both the uncoated and coated waveguides were tested in tailings pond water samples to determine the effects of water chemistry on the apparent dielectric

constant and waveform interpretation. Figure 5.5 compares the waveforms produced in tailings pond water and distilled water by an uncoated probe. The tailings pond water has a high cation ion concentration and high electric conductivity which significantly attenuates the signal, creating difficulties in determining the reflection point in the waveform. Therefore, the potential for errors in calculating the probe's apparent length (La) is significantly increased.

The dielectric material on the center rod of the coated probe reduces signal attenuation thereby making analysis more reliable in highly conductive media. Figure 5.6 illustrates that although some degree of signal attenuation occurs due to the chemistry of the tailings pond water, the apparent probe length (La) is very similar in both distilled water and tailings pond water. From this analysis, it was concluded that distilled water should not be utilized as a substitute for pond water in MFT calibrations. The use of tailings pond water in the calibration process enables the effects of signal attenuation to be studied over the range of volumetric water contents of interest. Therefore, it was concluded that coated TDR waveguides would be utilized for the MFT calibration process to minimize potential errors in waveform analysis.

Figure 5.7 illustrates a comparison of the apparent dielectric constants (coated waveguides) of tailings pond water and freeze / thaw effluent with distilled water at room temperature. Insufficient vacuum decant was collected to fully submerse the coated probe, therefore, Ka could not be accurately measured. This figure demonstrates that the apparent dielectric constant is relatively independent of water chemistry.

The effects of water chemistry on the apparent dielectric constant were further explored by measuring the variation of Ka with pH (Figure 5.8) utilizing coated waveguides. Sodium hydroxide was added to distilled water. Ka was calculated over a pH range of approximately 7.0 - 11.0. The results of this test indicate that

Ka is relatively insensitive to pH over this range. The variation in pH found in the tailings, pond water and effluent ranged from 7.4 - 8.9. Therefore, pH was not considered to significantly affect results.

From the initial analysis of distilled water and tailings effluent it was been concluded that, for dielectric coated TDR probes, the effects of temperature, water chemistry, and pH are relatively insignificant.

5.4 MFT calibration

MFT calibrations were conducted utilizing the protocol outlined in chapter 4. A total of eight calibration runs were performed for this project utilizing a combination of freeze / thaw dewatered MFT, vacuum decanted MFT, tailings pond water, and freeze / thaw effluent. Insufficient vacuum decant remained after the chemical analysis to be utilized in the calibration process. Table 5.3 summarizes the 8 calibration runs performed. Calibration run numbers 1, 2, and 3 were rejected due to erroneous results attributed to early attempts at developing a TDR probe calibration protocol and / or errors in waveform interpretation related to software development. By the fourth calibration run, these problems had been resolved. The data from the remaining five calibration runs are illustrated in Figure 5.9.

The initial calibrations were plotted as apparent dielectric constant versus percent solids (Figure 5.10). This was done under the assumption that the MFT was fully saturated, therefore, the volumetric water content (θ_v) could be calculated as a function of water content (w), solids content (S), dry density (ρ_d) and specific gravity (Gs).

$$w = (1- S) / S \qquad (5.1)$$

$$\rho_d = Gs / (1 + w\ Gs) * \rho_w \qquad (5.2)$$

$$\theta_v = w\ \rho_d \qquad (5.3)$$

The MFT calibration curve for Ka versus volumetric water content is illustrated in Figure 5.11 with Gs = 2.44. For comparison, Figure 5.12 was plotted utilizing Gs = 2.55 which is considered a typical value for the specific gravity of MFT. Figure 2.13 illustrates the effects of altering the specific gravity on the calibration curve by superimposing the data calculated with a Gs = 2.44 and Gs = 2.55 . From this figure it can be concluded that variations in specific gravity within the tailings pond will cause a small degree of error in the estimated volumetric water content. This error appears to be less than 1% for volumetric water contents greater than 65 %.

The MFT calibration may also be represented as the refractive index (N) versus volumetric water content. The refractive index is the square root of the dielectric constant and is calculated utilizing the following formula:

$$N = La / (Vp\ L) \qquad (5.4)$$

Figures 5.14 and 5.15 represent the calibration curves for specific gravity values of 2.44 and 2.55. The error due to variation in specific gravity (Figure 5.16) is similar to that for Ka measurements (Figure 5.13).

It is advantageous to develop calibration curves utilizing the refractive index versus apparent dielectric constant due to the linear relationship between refractive index and volumetric constant. By measuring the refractive index, it is possible to develop a two point calibration curve utilizing the refractive index of the pore fluid (θ_v = 100 %) and the refractive index of the dry soil mass (θ_v = 0). The simplicity of the calibration makes this an attractive option to the traditional 3rd order polynomial calibration curve stated in Topp et al. (1980) and discussed in section 2.4.4.1.

5.5 Effects of cable length

The effects of signal attenuation due to cable length was also explored. As stated in section 3.4, preliminary testing was conducted to determine the maximum practical cable length for use in the field. Tests were conducted on coated Soilmoisture waveguides (20 cm) submersed in distilled water with total cable lengths of 2 m, 12 m, and 22 m (Figure 5.17). This test demonstrates that cable lengths of 22 m will not cause significant error. However, similar testing in air (Figure 2.18) demonstrates significant signal attenuation. These findings are in agreement with Heimovaara (1993) who concluded that practical cable length for a given geometry was governed by readings in dry soil. Hence, longer cables can be utilized in high water content soils then in low water content soils.

All calibrations procedures for this project were performed with RG 58 cable. The use of other coaxial cable types should be explored to compare signal attenuation. Cable types recommended for further investigation include RG 213, RG 8 (type 9913), and RG 6.

5.6 TDR system accuracy

The data presented in Figures 5.11 through 5.15 indicates an accuracy ±2 % for the TDR unit (Tektronix 1502C) and the waveform analysis software utilizing 20 cm coated waveguides. The accuracy of this system is a function of the ability of the waveform analysis software to determine the reflection point at the end of the TDR waveguides. Therefore, the accuracy will be affected by the following factors: cable length, waveguide length, and pore fluid chemistry.

As stated in section 5.5 and illustrated in Figure 5.17, the coaxial cable will cause signal attenuation. This decrease in signal magnitude will result in a decrease in accuracy with which the reflection point at the end of the waveguides can be determined. The effects of signal attenuation do not appear to significantly affect results for cables less than 20 meters in length.

A decrease in the reflected signal strength causing a decrease in the accuracy with which the reflection point can be determined can also be attributed to waveguide length. Extremely long waveguides (> 50 cm) may cause a significant decrease in the magnitude of the reflected signal due to signal attenuation along the length of the waveguides.

The use of very short waveguides (< 10 cm) may also result in a decrease in accuracy when utilizing a Tektronix TDR unit. If the TDR signal travel time (Figure 3.1) is very short (< 1.0 nsec), then a significant decrease in accuracy may occur due to the limited resolution of the TDR oscilloscope.

Pore fluid chemistry can also attenuate the reflected signal if the conductivity of the pore fluid is sufficiently high. Figure 5.5 (pond water) illustrates signal attenuation in highly conductive media. This decrease in the magnitude of the reflected signal may result in significant error in determination of the reflection point at the end of the TDR waveguide.

Table 5.1 - MFT analysis report.

Salinity		
pH		8.9
Electrical Cond.	dS/m	2.97
Soluble Salts		
Calcium	meq/L	0.23
Magnesium	meq/L	0.15
Sodium	meq/L	31.3
Potassium	meq/L	0.22
Sulphate - S	meq/L	3.19
Chloride	meq/L	15.8
Carbonate	meq/L	1.98
Bicarbonate	meq/L	10.7
Soil Organics		
Oil	%	1.84
Water	%	66.1
Solids	%	32.0

Table 5.2 - Water analysis report.

		Tailings Pond	Freeze/Thaw	Vacuum Decant
pH		7.42	8.16	8.33
Electrical Cond.	uS/m	3000	2850	2870
Calcium	mg/L	5.2	3.6	4.8
Magnesium	mg/L	4.1	5.0	3.9
Sodium	mg/L	719	742	759
Potassium	mg/L	10.5	11.6	9.0
Iron	mg/L	0.5	4.0	0.0
Manganese	mg/L	0.02	0.03	0.01
Sulphate	mg/L	146	12.4	33.4
Chloride	mg/L	441	337	312
Bicarbonate	mg/L	925	1300	1310
T Alkalinity	mg/L	758	1070	1090
Hardness	mg/L	29.8	29.4	27.9
T. Dis. solids	mg/L	1780	1750	1770

Table 5.3 - Calibration summary.

Run #	Dewatering method	Effluent type
1	freeze / thaw	pond
2	freeze / thaw	pond
3	freeze / thaw	pond
4	freeze / thaw	pond
5	freeze / thaw	pond
6	freeze / thaw	freeze / thaw
7	freeze / thaw	freeze / thaw
8	vacuum decant	pond

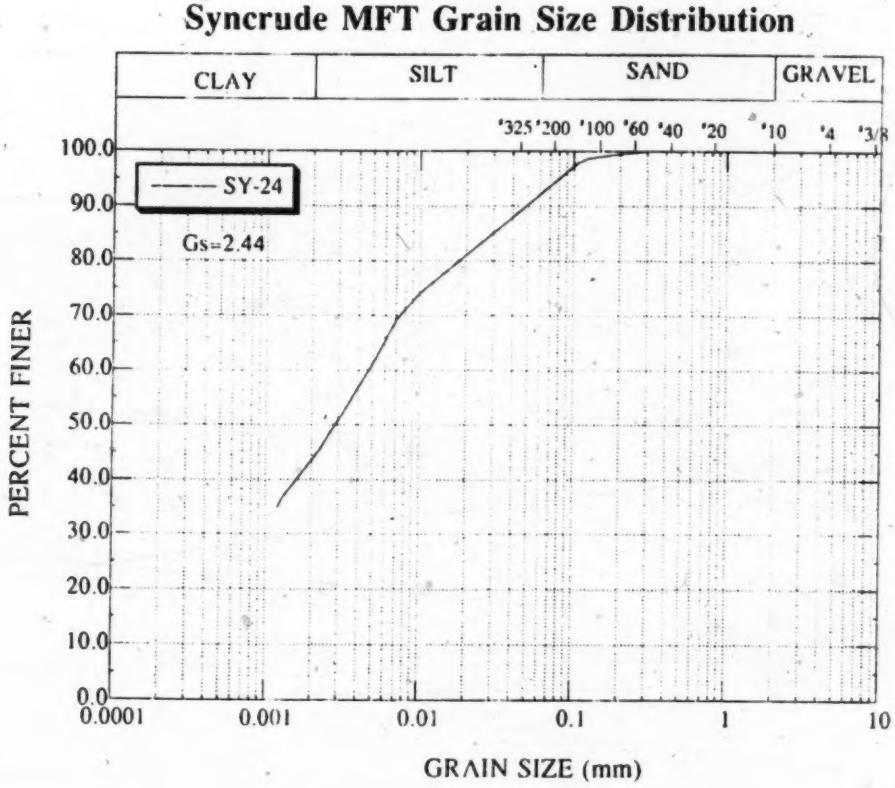


Figure 5.1 Grain size distribution (hydrometer analysis) for MFT barrel SY-24.

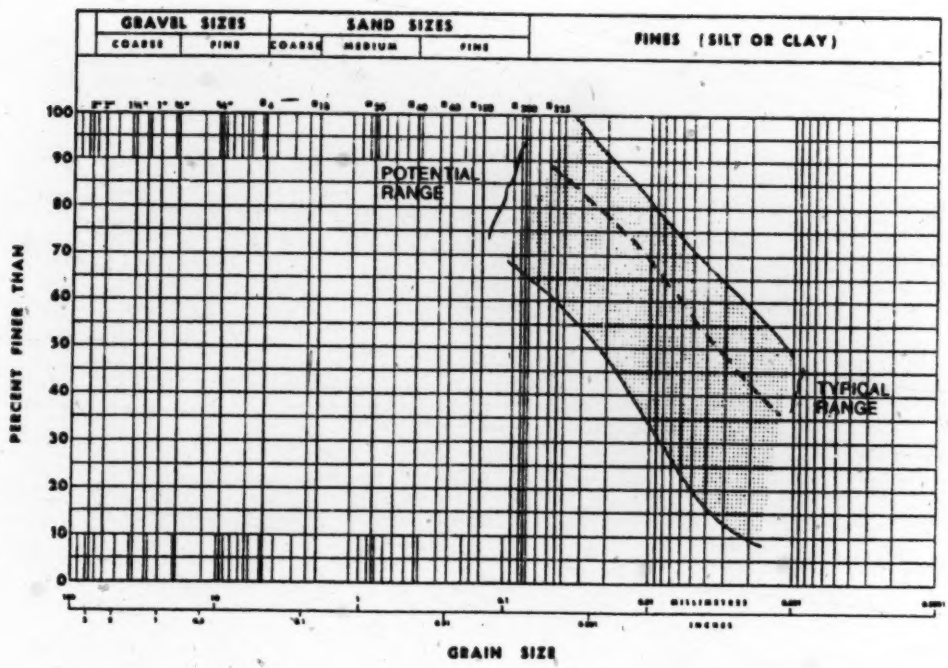


Figure 5.2 Syncrude MFT grain size distribution (modified from FTFC, 1995).

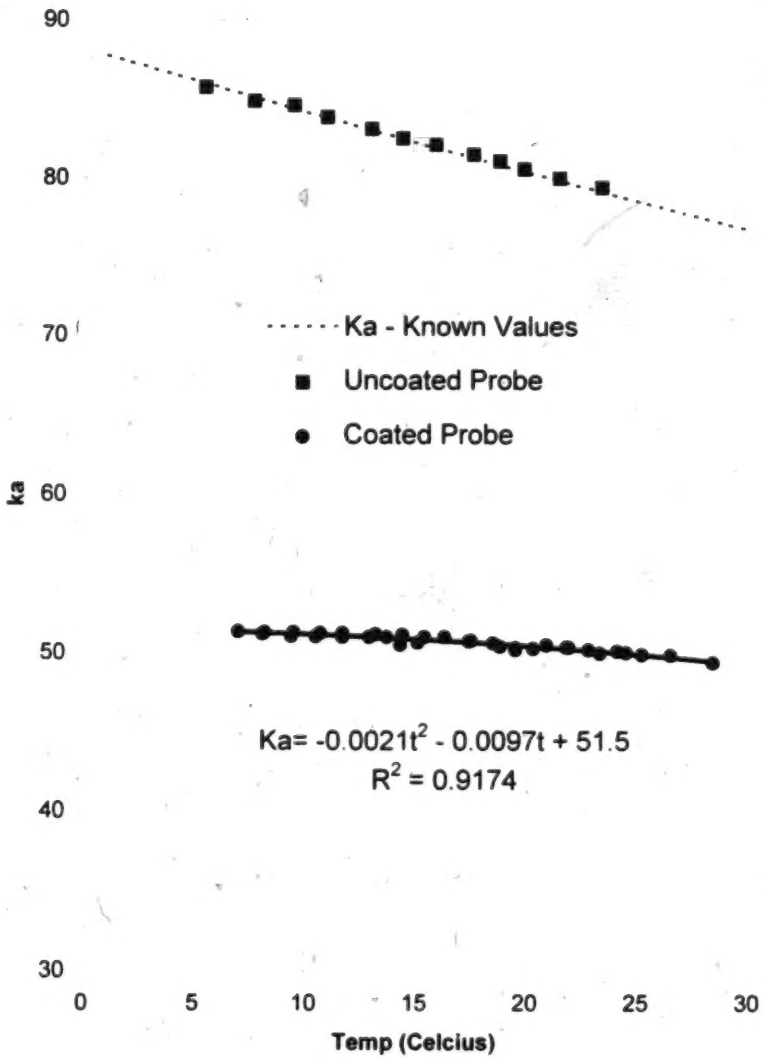


Figure 5.3 Temperature versus apparent dielectric constant of distilled water.

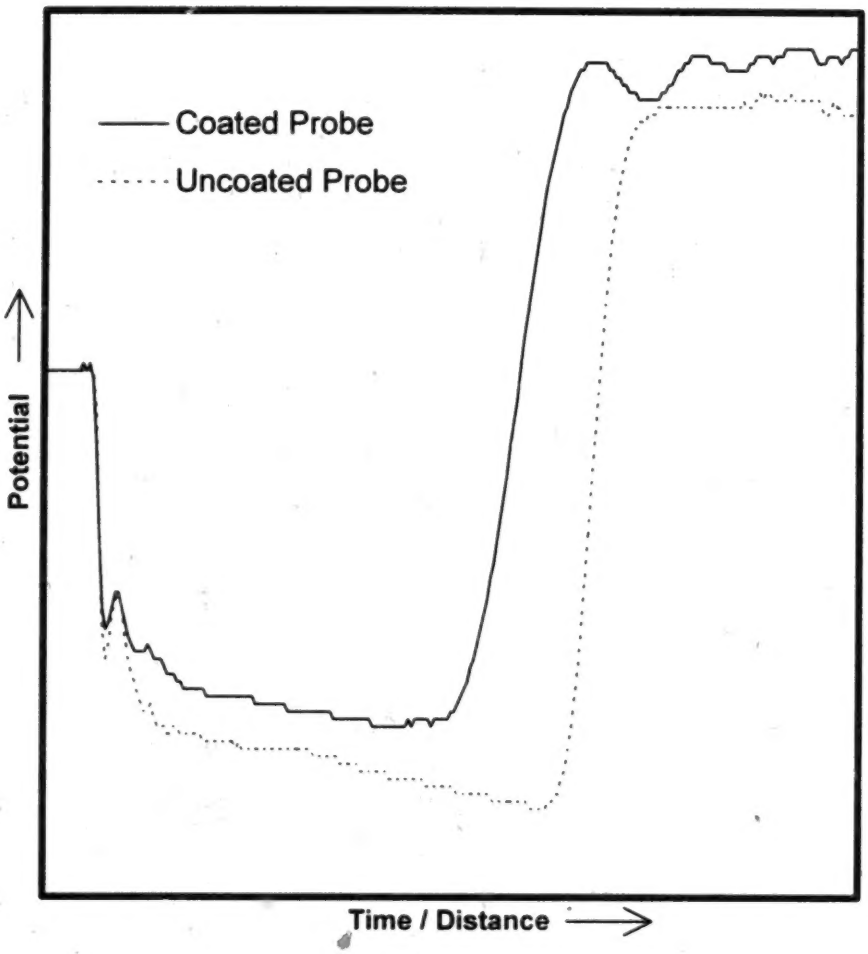


Figure 5.4 Uncoated versus coated waveguides (20 cm) in distilled water.

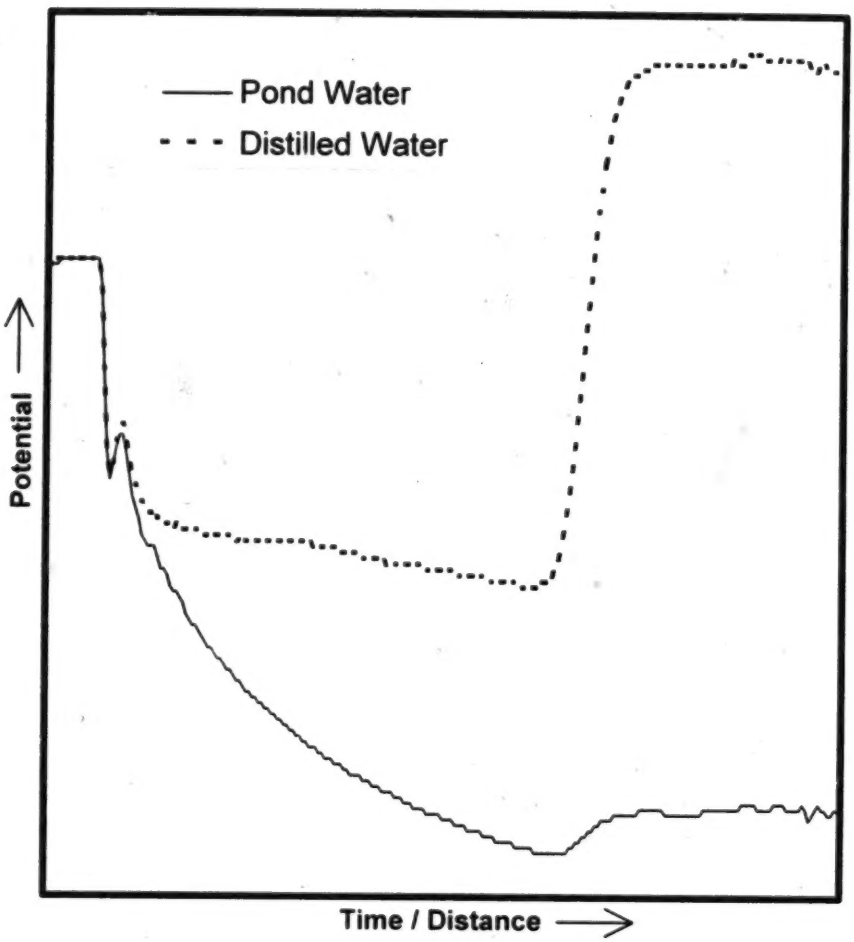


Figure 5.5 Uncoated probe (20 cm) in pond water and distilled water.

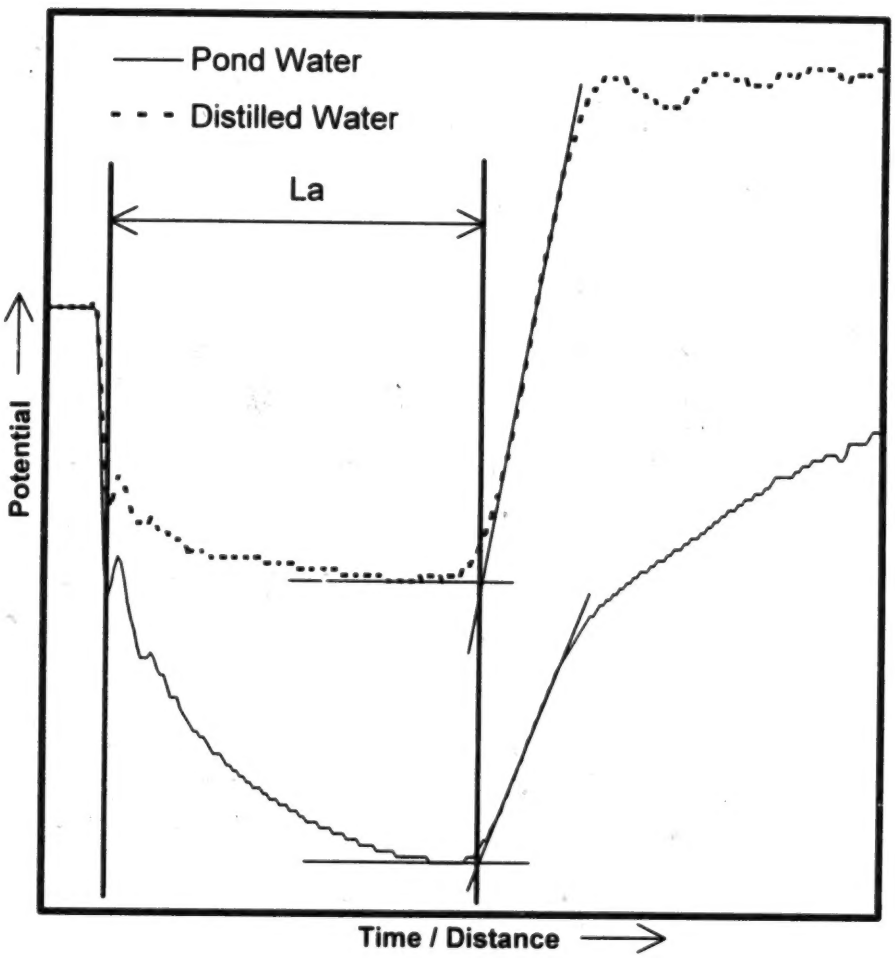


Figure 5.6 Coated probe (20 cm) in pond water and distilled water.

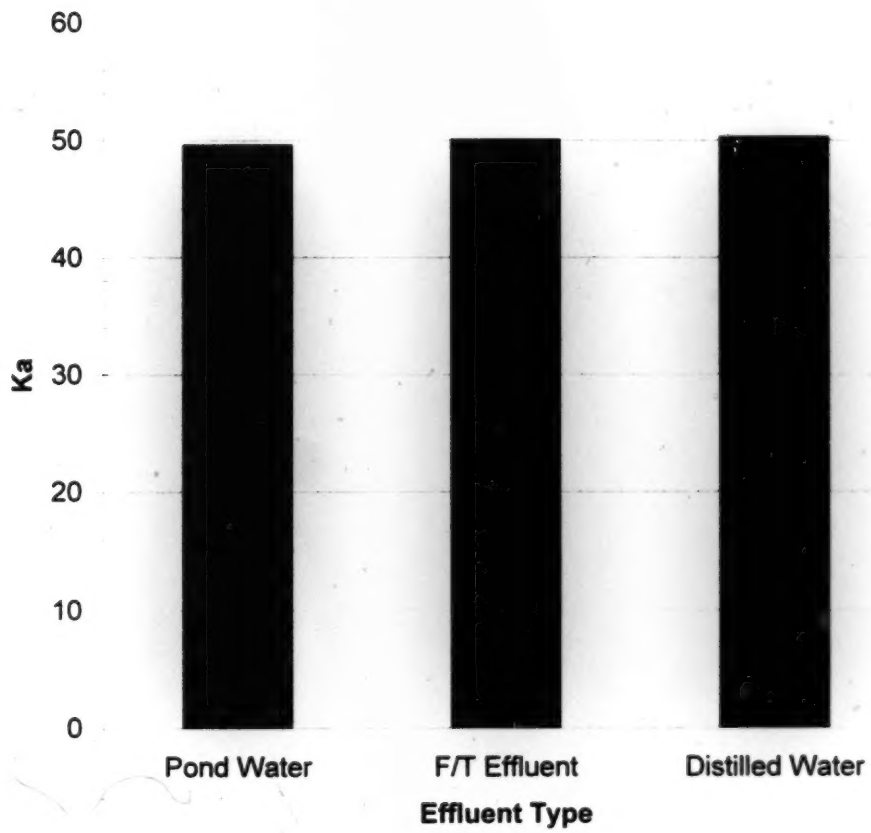


Figure 5.7 Comparison of the apparent dielectric constant (Ka) for pond water, freeze / thaw effluent, and distilled water for coated waveguides.

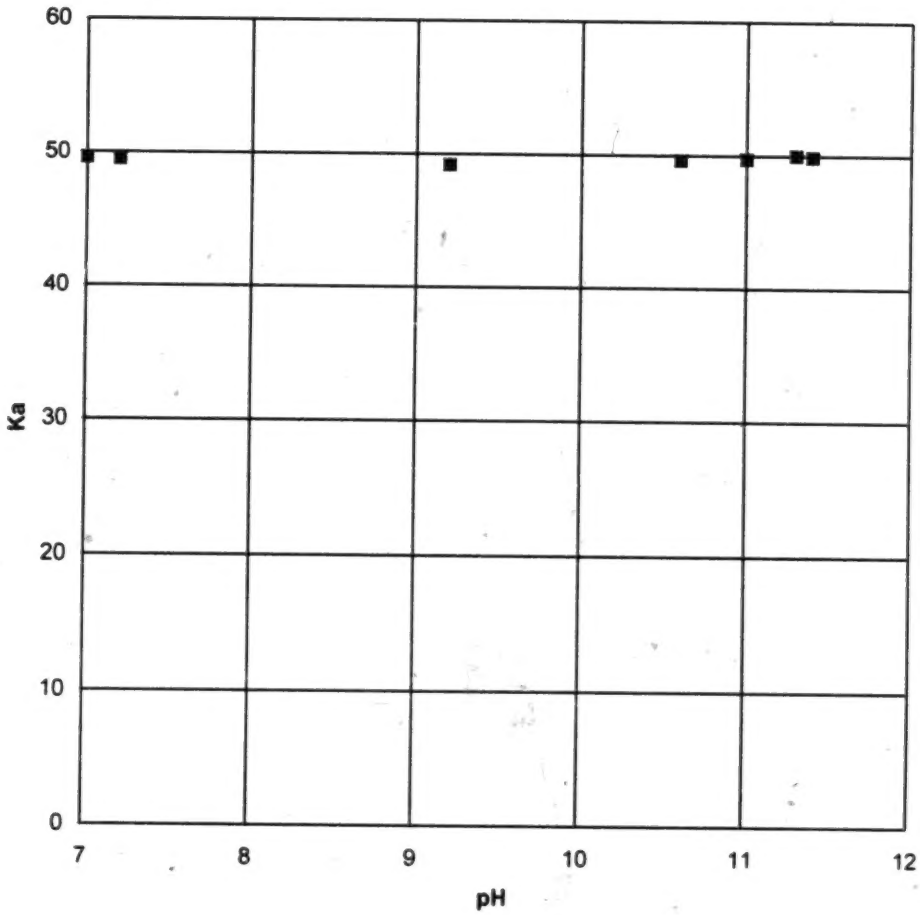


Figure 5.8 Apparent dielectric constant versus pH measured with a coated (20 cm) probe.

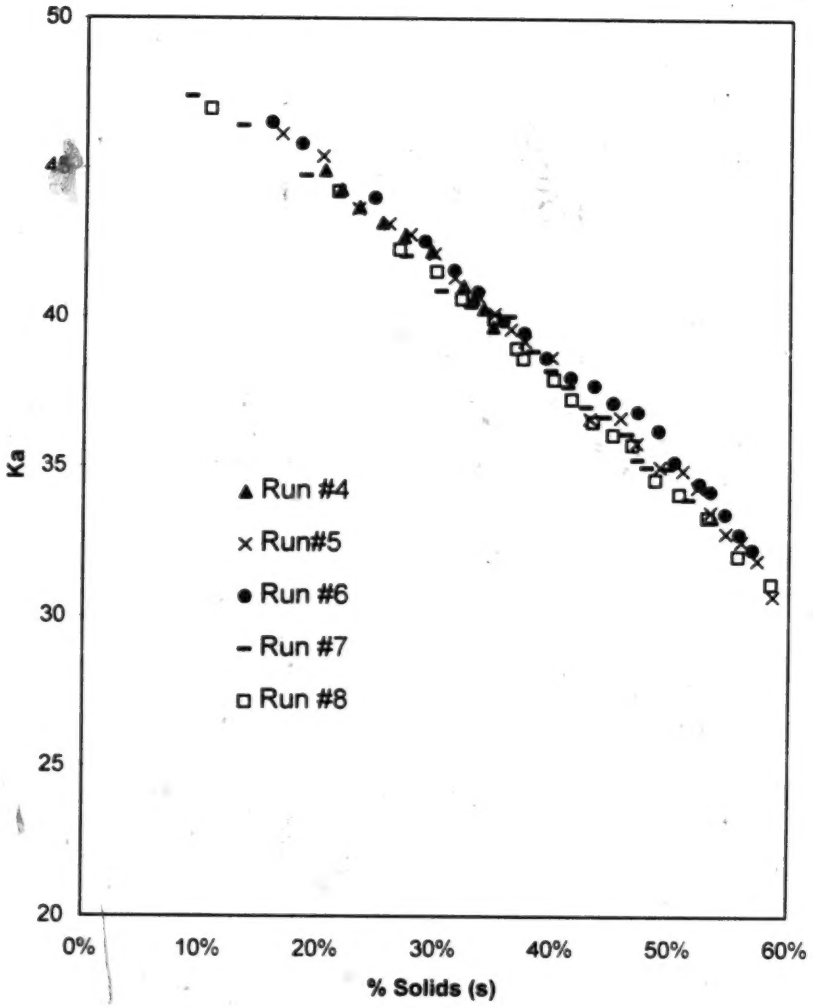


Figure 5.9 Apparent dielectric constant versus percent solids for 20 cm coated probe.

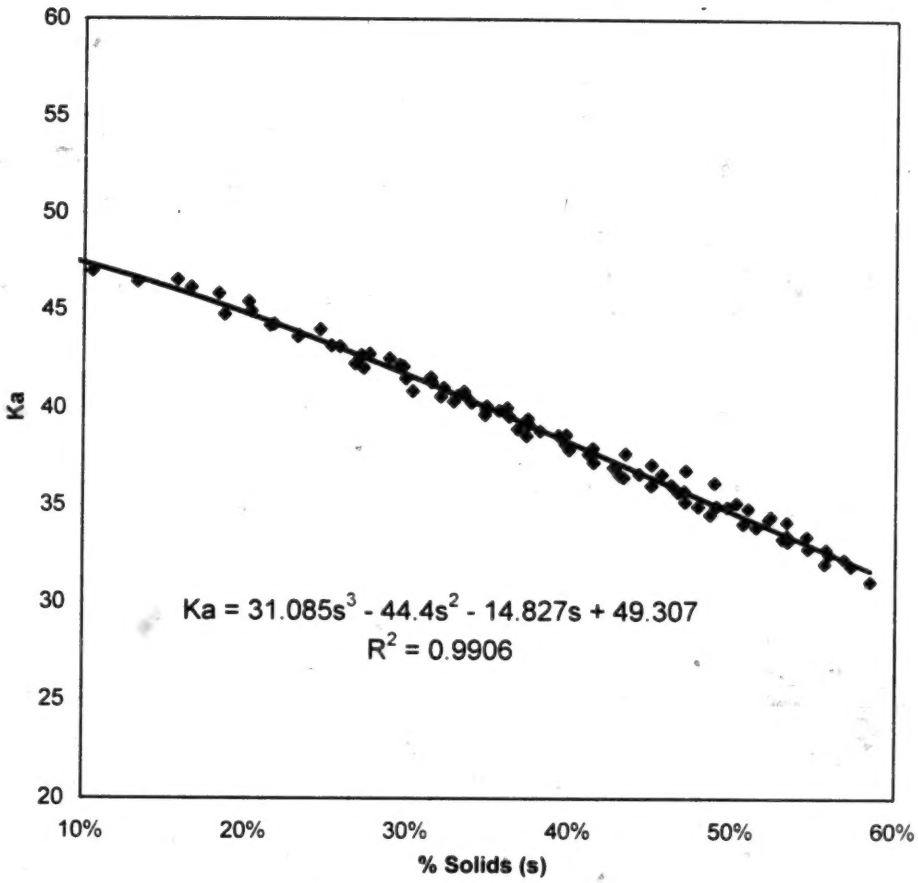


Figure 5.10 Apparent dielectric constant versus percent solids - calibration equation for 20 cm coated probe.

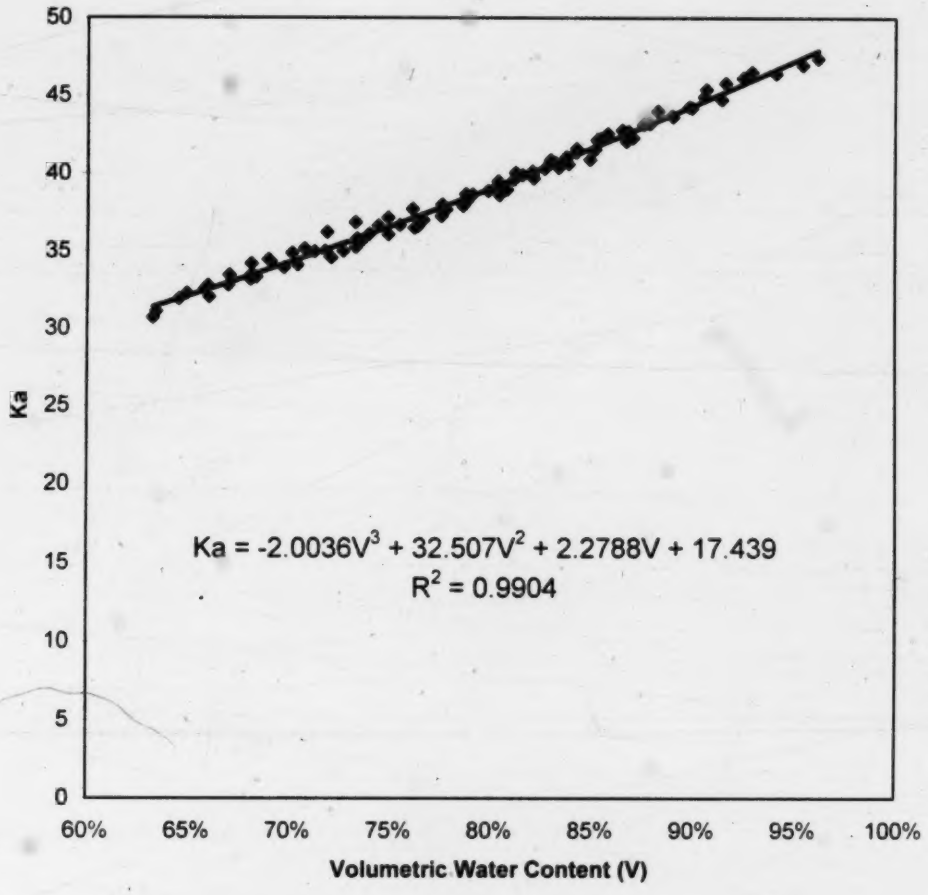


Figure 5.11 Apparent dielectric constant versus volumetric water content (Gs = 2.44).

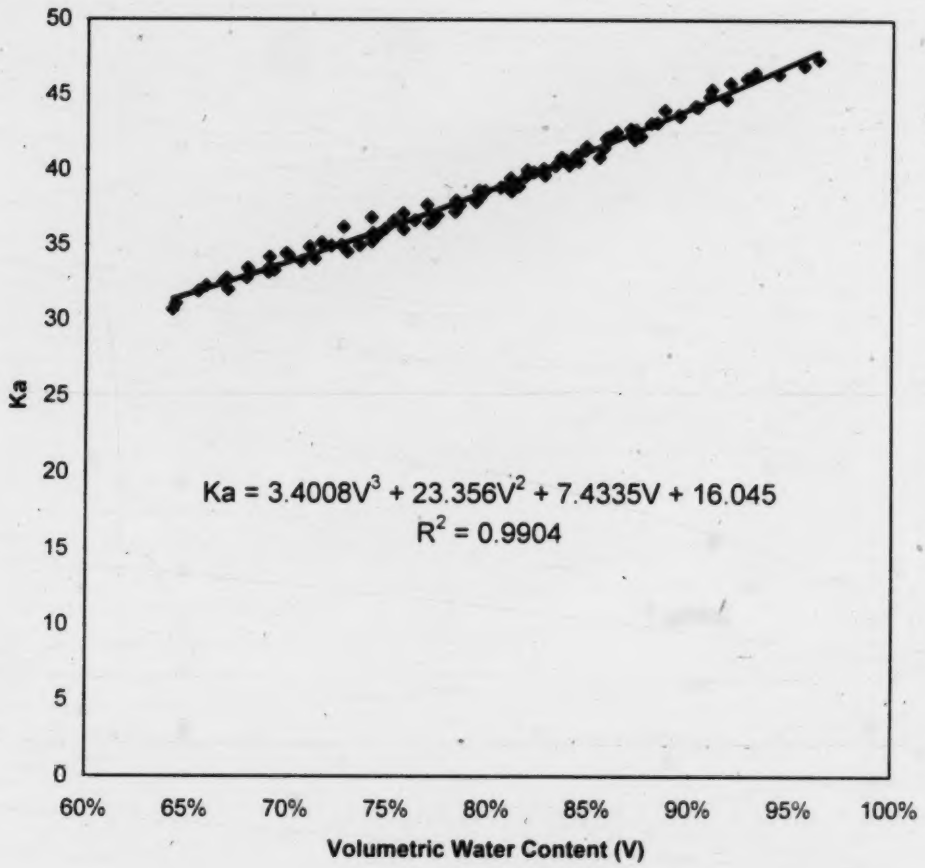


Figure 5.12 Apparent dielectric constant versus volumetric water content (Gs = 2.55).

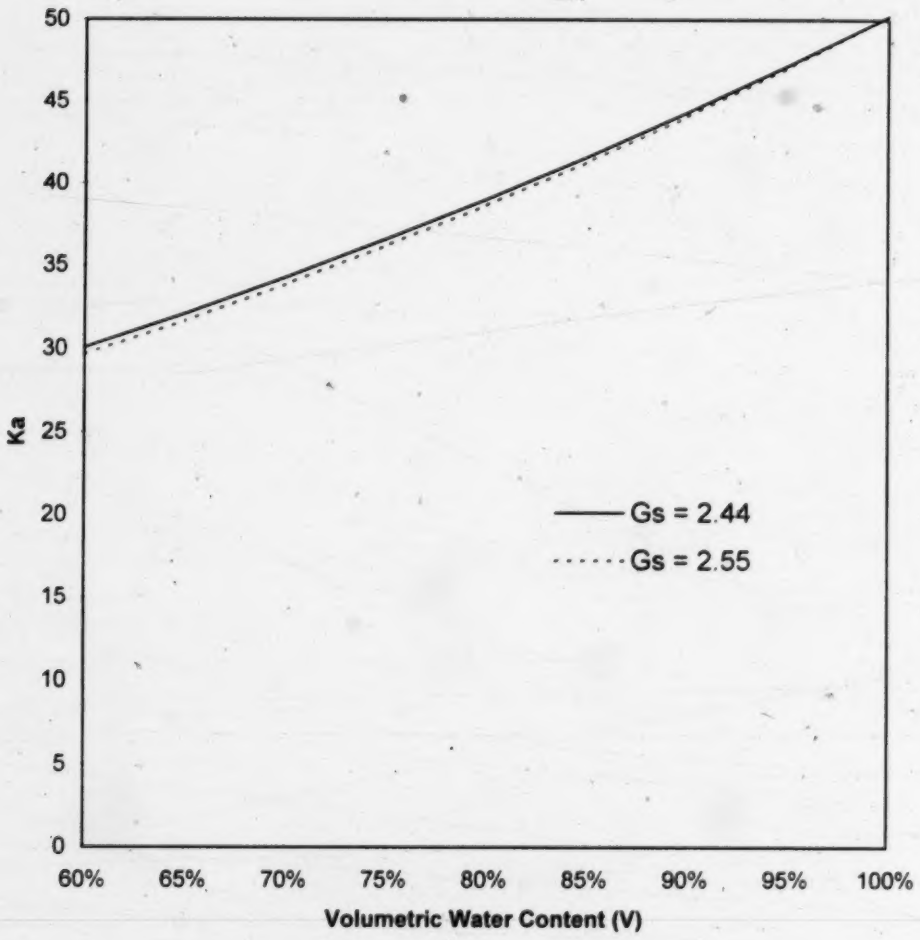


Figure 5.13 Comparison of apparent dielectric constant versus volumetric water content for a theoretical variation in specific gravity.

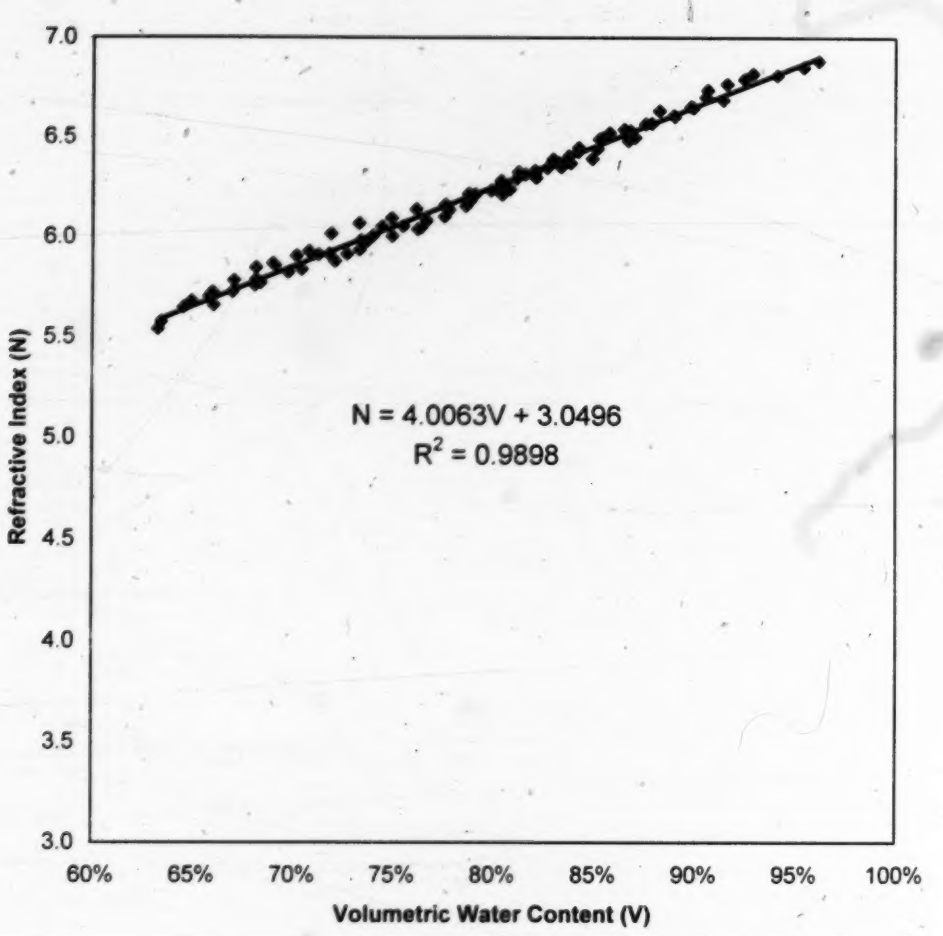


Figure 5.14 Refractive index versus volumetric water content (Gs = 2.44).

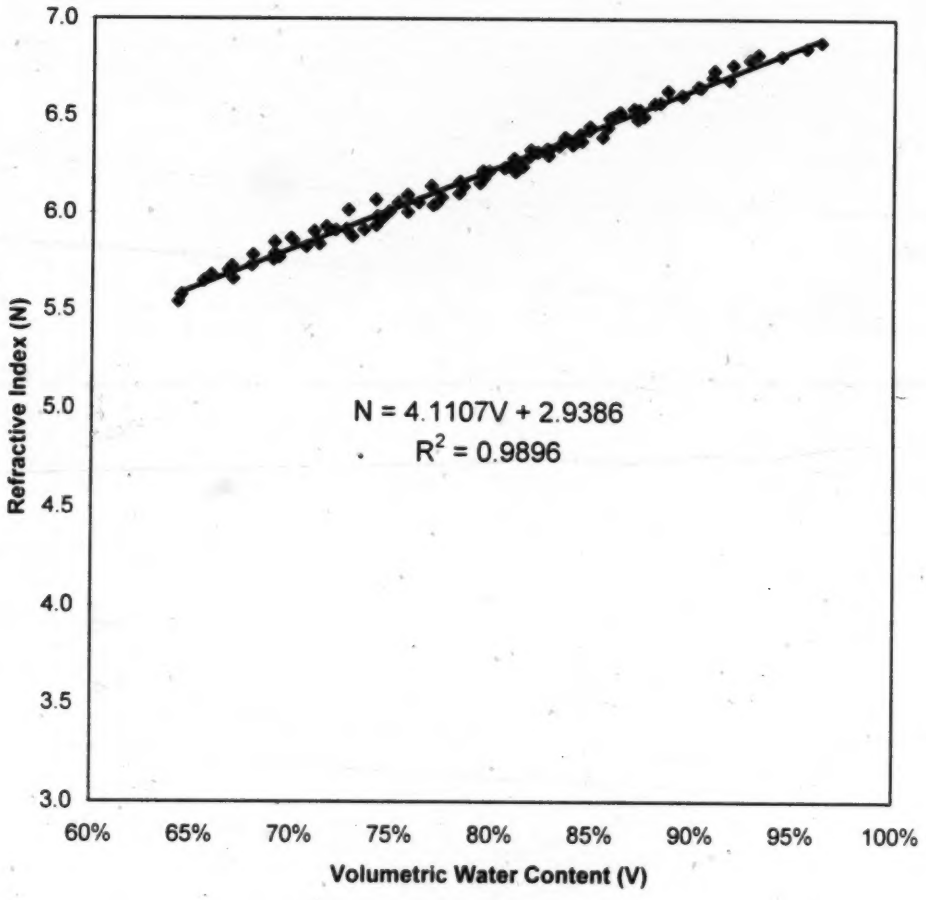


Figure 5.15 Refractive index versus volumetric water content (Gs = 2.55).

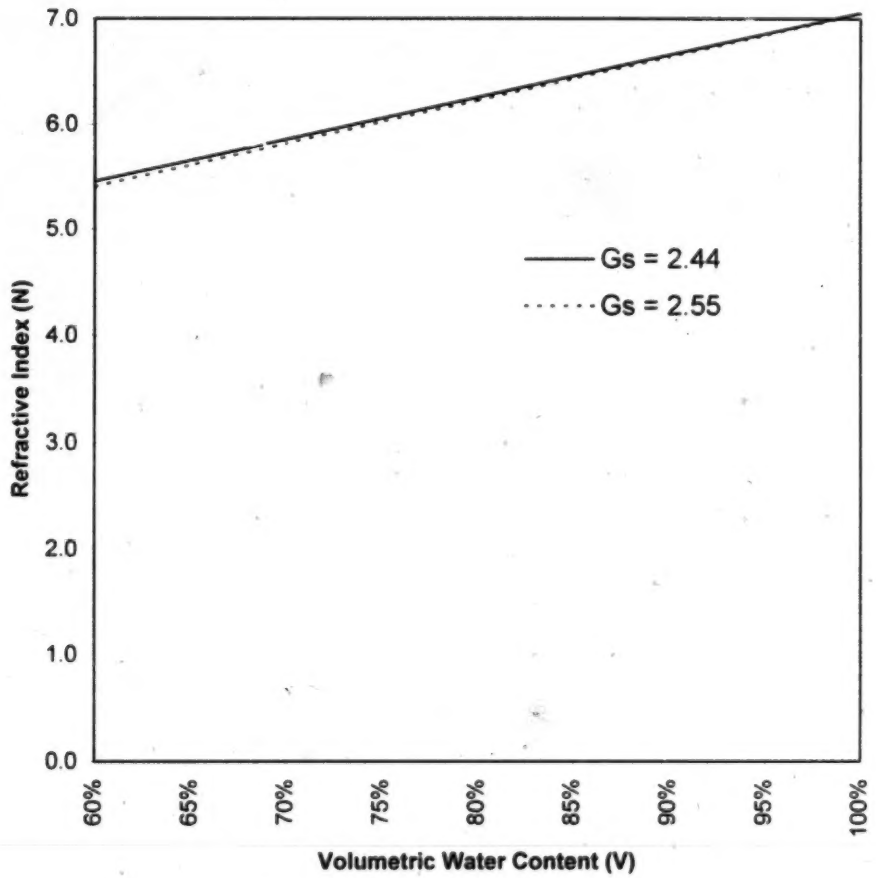


Figure 5.16 Comparison of refractive index versus volumetric water content for a theoretical variation in specific gravity.

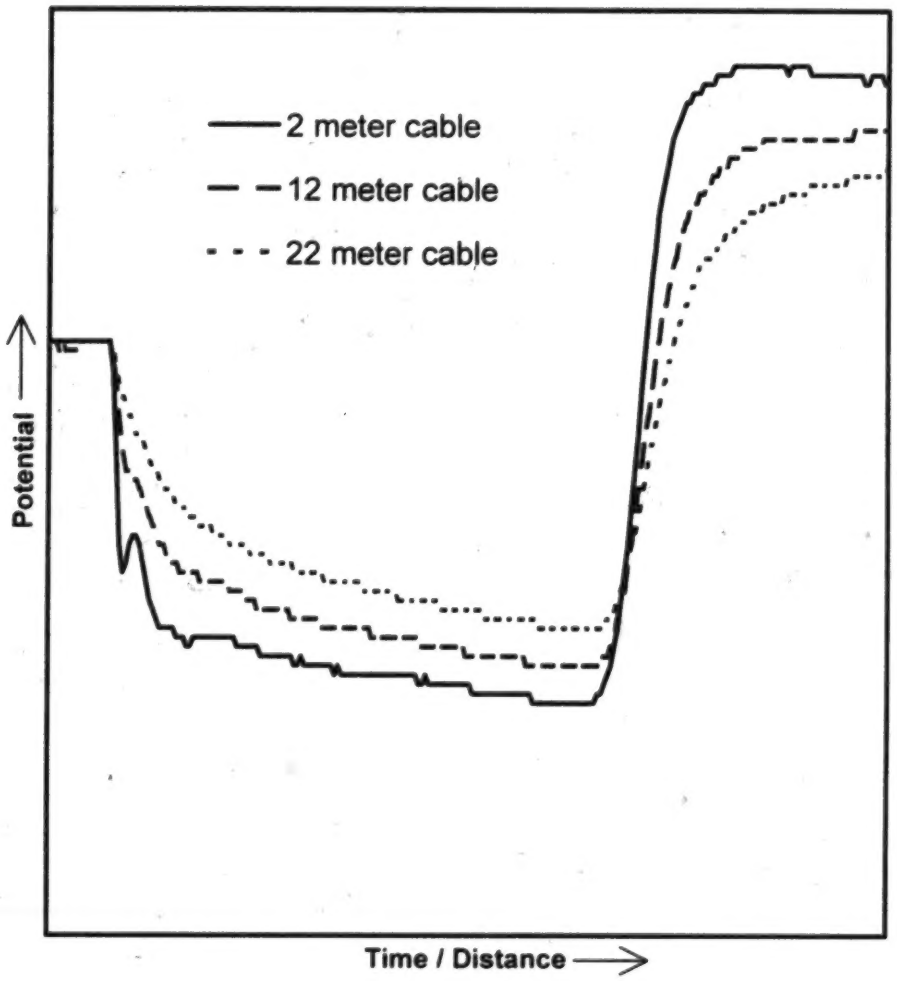


Figure 5.17 Effect of cable length in distilled water - 20 cm uncoated waveguides.

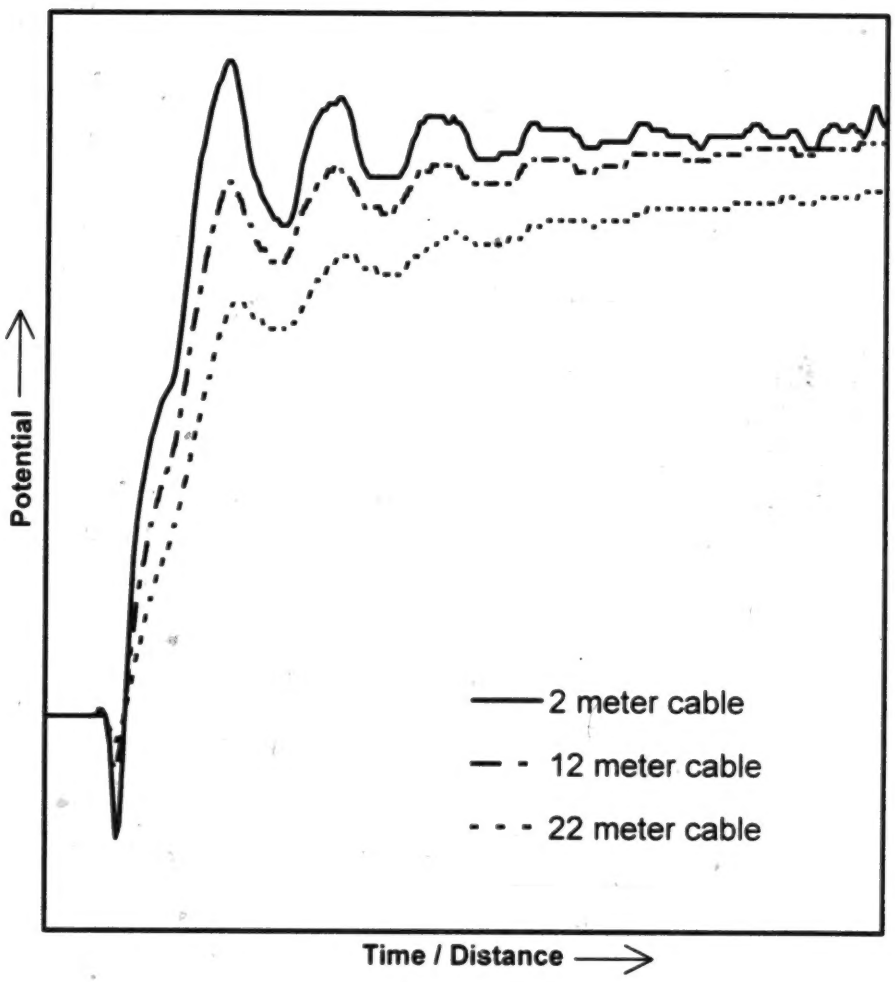


Figure 5.18 Effect of cable length in air - 20 cm uncoated waveguides.

Chapter 6

TDR Field Probe Development

6.1 Introduction

As stated in section 3.3, laboratory calibrations were performed with 20 cm commercial waveguides with a geometry similar to that illustrated in Figure 2.20. Although this probe geometry can be readily adapted for laboratory testing of low solids content MFT, it is not a practical design for geotechnical field applications. The main problem inherent with this probe geometry is the tendency for the waveguides to bend or break when inserted into a soil with solids contents greater than 70%. Another difficulty with the aforementioned geometry is related to adapting the waveguides to be lowered into a tailings pond of considerable depth.

The intent of this chapter is to suggest several possible probe geometries which could potentially be adapted for geotechnical field applications. In order to design an adequate TDR field probe it was necessary to develop a set of initial design parameters. The following guidelines were adopted for the design process:

- ⇒ the probe must be adaptable to being pushed into the ground by a drill rig or similar device;
- ⇒ the probe must be sufficiently robust to be pushed into stiff soils;
- ⇒ the waveguides must be resistant to deformation, therefore permitting repeated use;
- ⇒ the waveguides must provide sufficient signal resolution to permit minimum cable lengths of 20 m.

6.2 Construction materials

Four probe prototypes were developed for field applications (Figures 6.1 - 6.4). All probes were constructed from copper or brass waveguides, PVC cylindrical stock, and RG 58 coaxial cable.

As illustrated in Figure 5.3, the dielectric material utilized as a coating on the coated waveguides significantly affects the measured apparent dielectric constant. Prior to commencing the field probe design process it was hypothesized that the PVC cylindrical stock would have a similar effect on the measured apparent dielectric constant of the soil media. Hence, an optimal probe design should be rugged and provide a stable TDR waveform, but should also minimize the effect of the PVC ($Ka_{(PVC)} \cong 3.3$).

6.3 Proposed probe geometries

Probe #1, shown schematically in Figure 6.1, utilizes a 3-wire waveguide wrapped around PVC cylindrical stock. The resulting TDR waveform (Figure 6.5) is readily analyzed and produces an apparent dielectric constant in water at 22°C of approximately 76.5. The true dielectric constant of water at this temperature is 79.6. Therefore, it can be concluded that the PVC has a minimal effect on the waveform. However, custom calibrations would be required to account for the effect of the probe design on the measured value of the dielectric.

Another disadvantage related to this probe geometry is the need for the probe to have a sufficiently large diameter to permit the waveguides to be longer than 10 cm, thereby enabling adequate coaxial cables lengths to be utilized. This problem could potentially be overcome by utilizing remote shorting diodes discussed in section 2.4.4.3.3 and by Hook et al. (1992).

Probe #2 (Figure 6.2) resembles a typical 3 - wire waveguide embedded into a PVC cylinder. This geometry permits any desired waveguide length to be utilized, hence,

cable length is not a primary concern. The principal disadvantage associated with this geometry is the effect of the PVC cylinder on the measured apparent dielectric constant. Figure 6.6 shows a typical waveform in water at 22°C. The measured apparent dielectric constant is approximately 42.4, which is significantly lower than the actual value of 79.6. The influence of the PVC cylinder effectively flattens the calibration curve and may potentially result in an decrease in accuracy for the determination of soil moisture content.

Probe #3 (Figure 6.3) was proposed by Yokuda and Smith (1993) and is illustrated in Figure 2.23. This geometry is unusual because it utilizes a 2 - wire waveguide which the authors claim did not require an impedance balancing transformer (balun). The TDR waveform (Figure 6.7) produced by this probe indicates that although the local minimum is more difficult to determine relative to a 3-wire probe, it is possible to calculate the end of waveguide reflection point with a high degree of repeatability.

The measured apparent dielectric constant of water at 22°C was approximately 42.5. This indicates that the PVC cylinder has a similar influence on probe #2 and #3. Therefore, the same disadvantages inherent with probe #2 also apply to probe #3.

Probe #4 (Figure 6.4) is a modification of probe #2 (3-wire waveguide geometry). The modification involved moving the outer waveguides to opposing positions at the center on the PVC cylinder. This was done to measure the effects of the PVC on the apparent dielectric constant of the soil media. Although probe #4 produces a very stable and easily interpreted waveform (Figure 6.8) the effect of the PVC is significantly increased relative to probe #2. The measured apparent dielectric constant of water at 22°C is approximately 36.4 versus the actual dielectric constant of 79.6.

6.4 Recommendation for field probe design

Probes #1 and #2 hold the greatest promise for success in the development of field TDR probes for geotechnical applications. Probe #1 requires a significantly larger diameter PVC cylinder (increase in diameter of approximately 1 cm) than probe #2, however, it also demonstrates significantly lower effects on the waveform due to the PVC material.

For application at Syncrude, where the solids contents are less than 80% for both the mature fine tailings (MFT) and combined tailings (CT), the increased probe diameter (probe #1) should not pose any significant foreseeable difficulties. Hence, this geometry is recommended for further development. The adaptation of remote shorting diodes should also be further explored to potentially decrease the required PVC cylinder diameter and increase the permissible cable lengths.

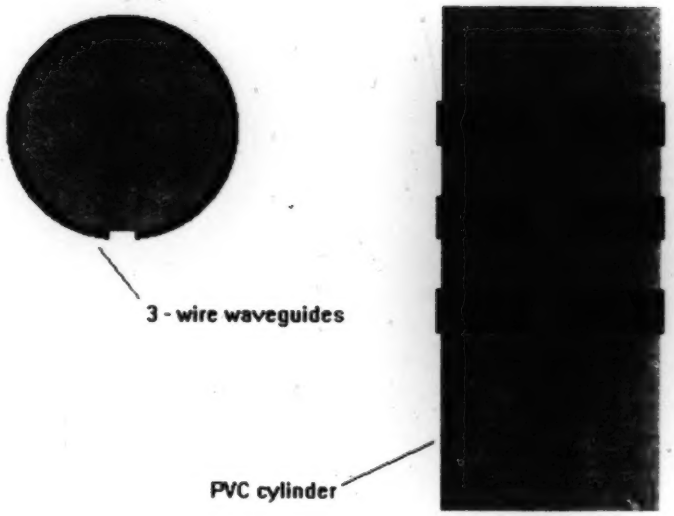


Figure 6.1 Probe geometry #1 (dimensionless).

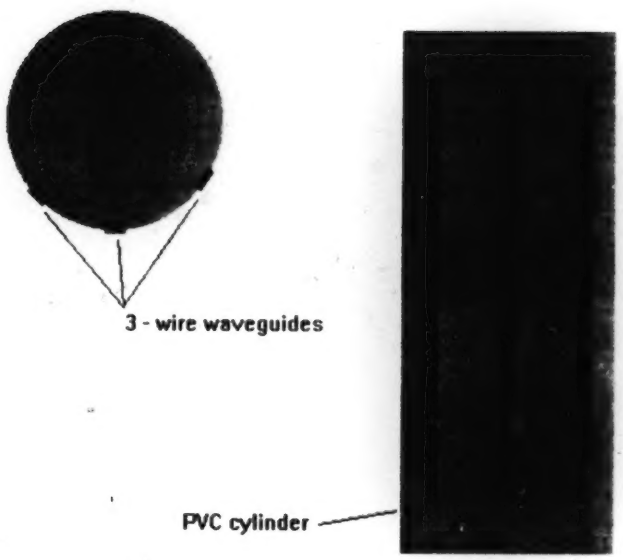


Figure 6.2 Probe geometry #2 (dimensionless).

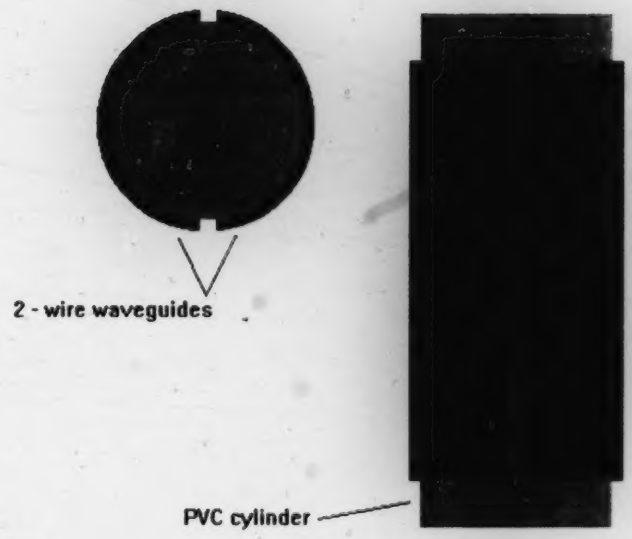


Figure 6.3 Probe geometry #3 (dimensionless).

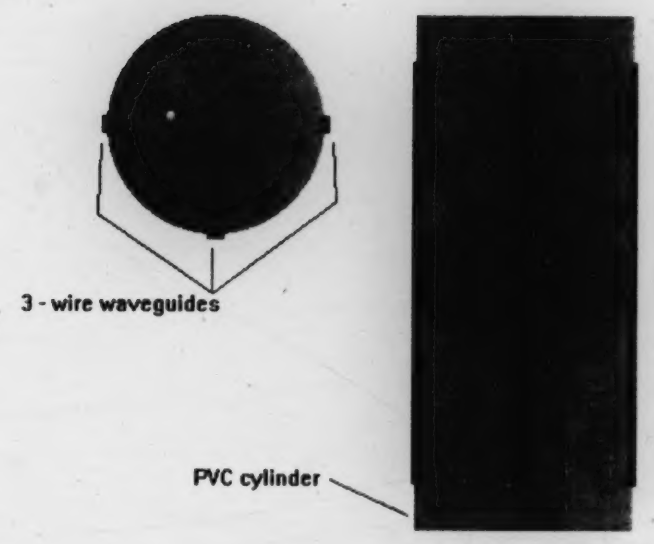


Figure 6.4 Probe geometry #4 (dimensionless).

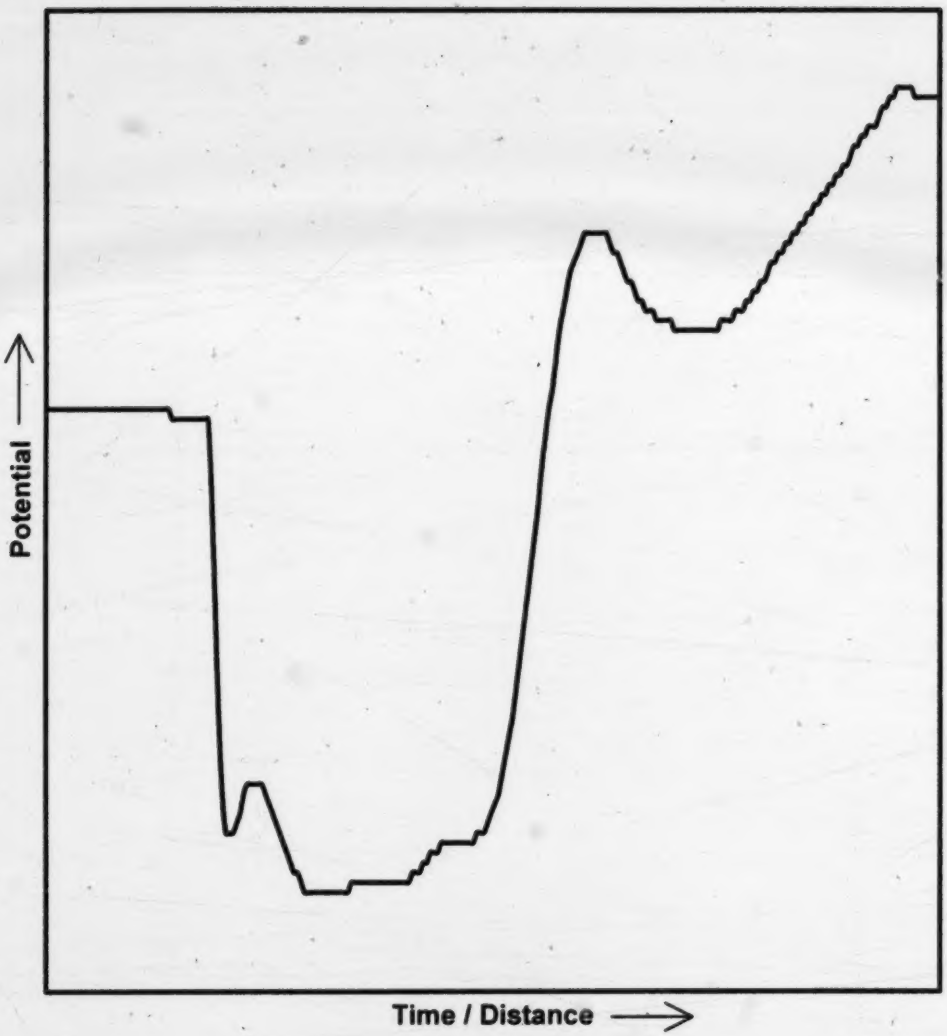


Figure 6.5 Probe geometry #1 waveform for water at 22°C.

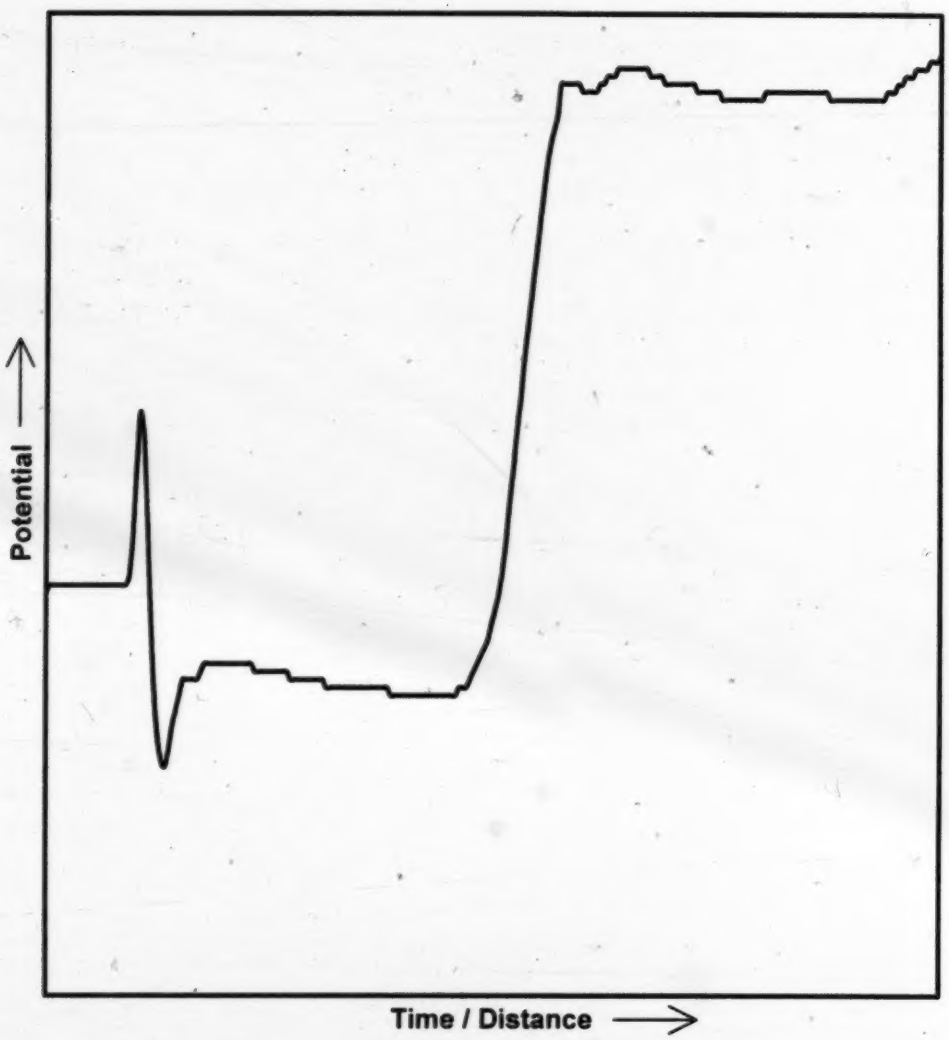


Figure 6.6 Probe geometry #2 waveform for water at 22°C.

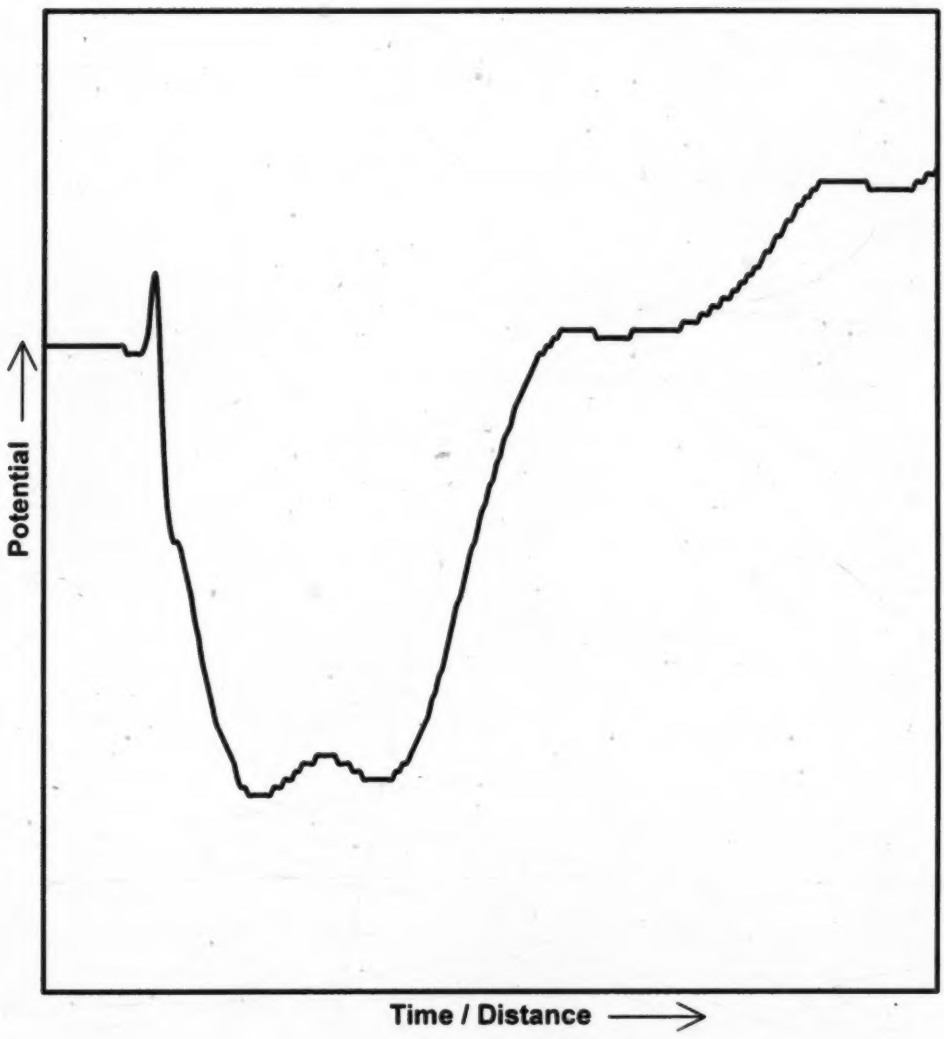


Figure 6.7 Probe geometry #3 waveform for water at 22°C.

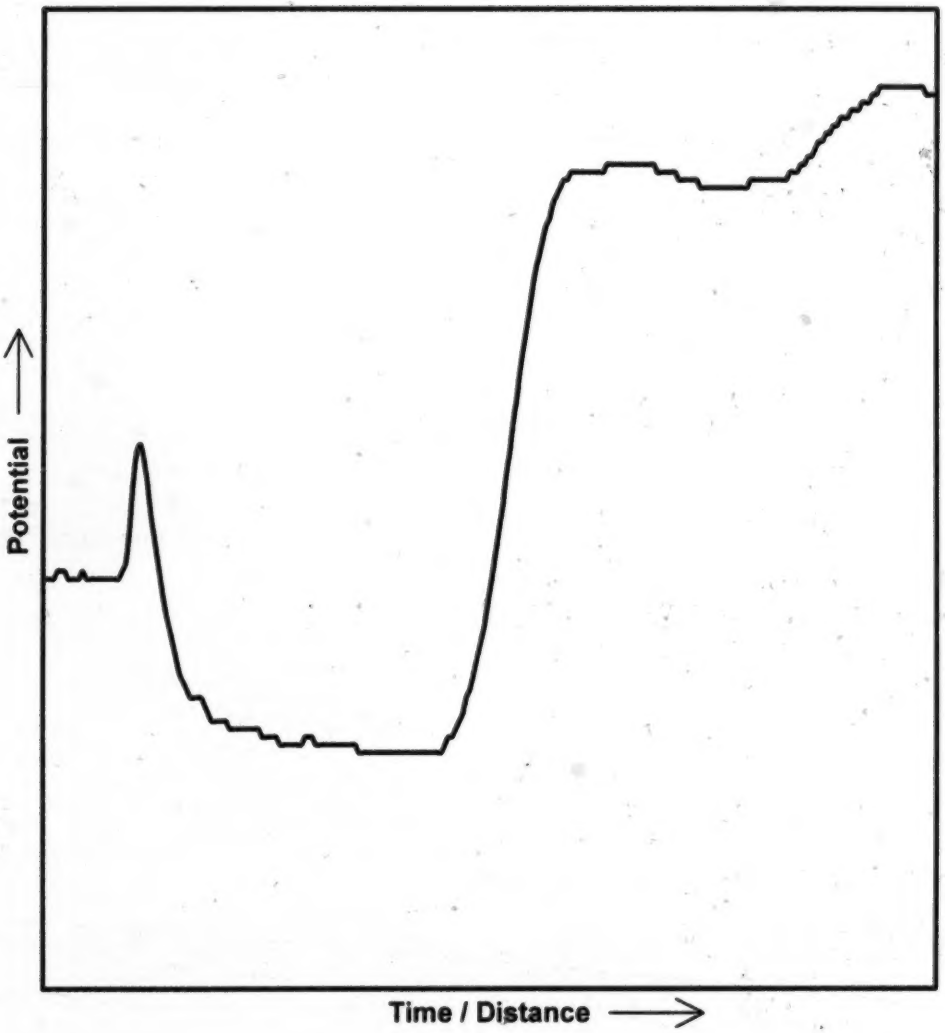


Figure 6.8 Probe geometry #4 waveform for water at 22°C.

Chapter 7

Conclusions and Recommendations

7.1 Overview

The objectives of this research project were successfully completed. The literature search examines several possible methods for determining the moisture content of soils. Due to the low solids contents (15% ≤ % solids ≤ 55%), high sodium concentrations and high levels of conductivity of Syncrude's mature fine tailings (MFT), measurement of the dielectric constant utilizing time domain reflectometry (TDR) was determined to be a suitable technology.

7.2 TDR calibration for MFT

The results of the preliminary calibrations presented in Chapter 5 confirm the observations made by Topp et al. (1980) that TDR measurements are essentially independent of temperature and salt concentration. Data presented in Chapter 5 also indicated an insensitivity to pH. Further research is required to investigate the possible effects of soil type (cohesive, non-cohesive, and organic) and density.

The most significant observation made regarding TDR calibrations for MFT: the validity of a two point calibration curve based on the refractive index (N) versus volumetric water content and the repeatability of Ka versus percent solids. Topp et al. (1996) states that a two point calibration curve (N versus θ_v) is applicable and produces results similar to the Topp equation presented in Topp et al. (1980) and discussed in section 2.4.4.1. This procedure greatly simplifies the calibration of TDR probes by utilizing a linear relationship between the refractive index (N) of the pore water and dry soil. A third data point is recommended to confirm the validity of the two point calibration.

Based upon the calibrations presented in chapter 5, it appears that time domain reflectometry is feasible for use in determining the volumetric water content of mature fine tailings. The volumetric water content may readily be converted to solids content if the soil is fully saturated and the specific gravity is known. As demonstrated in section 5.4, a reasonable estimate of the specific gravity (± 5%) does not result in significant error in the estimated solids content.

7.3 Software development

M-TDR version 1.1 was developed to be a "user friendly" software package which is capable of waveform analysis for most conceivable waveguide geometries. This software package was tested extensively in the laboratory and performed satisfactorily. An operating manual is included in Appendix A. Further modifications are recommended to include conductivity analysis capabilities.

7.4 Field probe development

Chapter 6 presented four potential waveguide geometries which could be adapted for use in a "push" technology field probe. A geometry similar to probe #1 is recommended for further development, however, probe #2 also holds considerable promise. A custom calibration curve is required for each of these probe geometries due to the influence of the PVC material on the measured apparent dielectric constant of the soil media.

7.5 Coaxial cable

Laboratory calibrations were conducted utilizing RG 58 coaxial cable. As stated in section 3.4 and confirmed by laboratory testing (section 5.5), cable lengths in excess of 20 m (RG 58) can be utilized without significantly attenuating the signal. Further testing should be conducted utilizing higher quality cables (as stated in section 5.5) or remote shorting diodes (Hook et al., 1992) to increase the operable cable length for field applications.

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